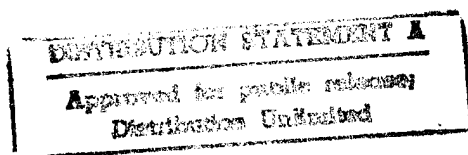


# REPORT

CONTRACT NO. F04606-89-D-0034/Q804

## IDENTIFICATION OF BIODEGRADABLE/ ENVIRONMENTALLY COMPATIBLE METHODS FOR EPOXY REMOVAL -- PHASE I



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To

THE AEROSPACE GUIDANCE AND

METROLOGY CENTER

NEWARK AIR FORCE BASE

August 6, 1993



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**SUMMARY REPORT**

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COMPATIBLE METHODS FOR EPOXY REMOVAL -- PHASE I**

to

**THE AEROSPACE GUIDANCE AND METROLOGY CENTER  
NEWARK AIR FORCE BASE**

**August 6, 1993**

by

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## EXECUTIVE SUMMARY

The Aerospace Guidance and Metrology Center (AGMC), located at Newark Air Force Base (NAFB), Ohio, repairs and maintains navigation and guidance equipment for the United States Air Force and other components of the Department of Defense. Thousands of these delicate and sophisticated electromechanical devices are repaired each year at the Center.

The repair of these devices may require removal of a variety of cured epoxy adhesives and coatings. Current methods of removing epoxy resins can cause surface damage to the parts, or may use chemicals which are toxic or carcinogenic, or which may be environmentally undesirable.

The objective of this first phase program was to identify alternative, solvent-based methods for epoxy removal which are efficient, more environmentally compatible than current processes, safe in their application, and which do not degrade the reliability of the equipment being repaired and maintained by AGMC.

A focused literature search and adhesive supplier contacts were used to define a preliminary set of candidate solvent systems, including commercially available products. A theoretical solubility parameter model was then used to screen the candidates.

Fourteen potential solvents and solvent systems were identified for preliminary feasibility testing. Swelling/cracking tests were carried out for one epoxy and the fourteen solvents. The three solvents showing the most potential (N-methyl pyrrolidone, benzyl alcohol and furfuryl alcohol) were chosen for epoxy removal tests. These tests were carried out for the three neat solvent systems and three solvent/water mixtures using aluminum coupons coated with seven different epoxies in common use at AGMC. All the tested epoxies were easily removed by one or more of the solvent mixtures after twenty-four hour exposure at 70 C. Epoxy removal tests with sample parts supplied by AGMC were carried out to confirm the performance of these solvent mixtures with realistic substrate materials and geometries. Parts testing confirmed the overall effectiveness of the three solvents.

Results of the first phase of the program are sufficiently promising to recommend that a second phase of the program be carried out to develop an alternate epoxy removal process based on the use of the more environmentally compatible candidate solvents identified above.

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## **SUMMARY REPORT**

### **IDENTIFICATION OF BIODEGRADABLE/ENVIRONMENTALLY COMPATIBLE METHODS FOR EPOXY REMOVAL -- PHASE I**

#### **1.0 INTRODUCTION**

##### **1.1 Background**

The Aerospace Guidance and Metrology Center (AGMC), located at Newark Air Force Base (NAFB), Ohio, repairs and maintains navigation and guidance equipment for the United States Air Force and other components of the Department of Defense. Thousands of these delicate and sophisticated electromechanical devices are repaired each year at the Center.

The repair of these devices may require removal of a variety of cured epoxy adhesives and coatings. Epoxy resins are widely used in the aerospace industry, both as base materials and as adhesives on electromechanical parts and components, because of their high temperature resistance, excellent strength and good resistance to chemicals. During the repair of the parts and components it is frequently necessary to remove the adhesive. Since the thermoset epoxy adhesive is crosslinked, it is difficult to dissolve. Current methods of removing epoxy resins involve the use of heat and force, mechanical scraping, stripping by oxidation and degradation, and bead blasting. Many of these operations, such as mechanical scraping or bead blasting, can cause surface damage to the parts. Other operations, such as chemical stripping, may use chemicals which are toxic or carcinogenic, or which may be environmentally undesirable.

AGMC is committed to reducing the use of hazardous chemicals and improving its repair processes. Therefore, the objective of this first phase program was to identify alternative, solvent-based methods for epoxy removal which are efficient, more environmentally compatible than current processes, safe in their application, and which do not degrade the reliability of the equipment being repaired and maintained by AGMC. Standards of comparison include current epoxy removal processes that use dimethyl sulfoxide (DMSO) and nitric acid, methyl ethyl ketone, methylene chloride, chlorofluorocarbons (CFC's), and mechanical scraping.

## **1.2 Approach**

This phase of the program included three technically-based tasks:

### **Task 1.0 -- Information Collection**

Information was collected from AGMC regarding parts of interest, current processes and criteria for development of a new epoxy removal process. A focused literature search was conducted, using selected databases, conference proceedings, and other related Air Force programs as information sources. Adhesive suppliers were contacted for recommendations. Candidate solvent systems, including commercially available products, were identified.

### **Task 2.0 -- Information Analysis and Process Selection**

Information assembled in Task 1.0 was reviewed to determine important solvent-epoxy interactions that could enhance swelling or crazing of the epoxy. A theoretical model, based on solubility parameter analysis, was used to further identify candidate solvent systems for testing with epoxies in use at AGMC. Fourteen potential solvents and solvent systems were identified for preliminary feasibility testing.

### **Task 3.0 -- Preliminary Feasibility Experiments**

Preliminary feasibility experiments were carried out to evaluate the potential of selected solvents and solvent systems for swelling, softening, and removing epoxy adhesives from typical component parts repaired by AGMC. The scope of the experiments was defined by: (1) epoxy adhesives currently in use by AGMC; (2) use of solvent systems chosen to be more environmentally compatible than those systems currently used by AGMC; and (3) use of solvent systems that allow washability of the residue, if any, using environmentally benign wash solvents or water.

Cured bulk epoxy samples and cured epoxy-coated, aluminum lap shear test coupons for seven different epoxy formulations were available from a previous AGMC study, and were used in this program. Swelling/cracking tests were carried out for fourteen solvent systems, using one bulk epoxy formulation to measure the amount of swelling induced by

each solvent system. The three solvents showing the most potential for swelling and cracking were chosen for epoxy removal tests.

Epoxy removal tests were carried out for the three neat solvents and three solvent/water mixtures using aluminum lap shear coupons coated with the seven different epoxies. A brief study of the effect of the use of acid rather than water as a hydrolyzing agent was also carried out.

Epoxy removal tests were performed for the two most effective solvent mixtures from the coupon tests, using six selected precision parts supplied by AGMC. The parts were chosen to provide several typical substrate metals. Compatibility of beryllium with the three solvent/water mixtures was also evaluated.

## **2.0 RESULTS**

Results from Task 1.0 include information collected from AGMC, results from the literature search, and information from adhesive and solvent suppliers. Results from Task 2.0 include a summary of analysis using a solubility parameter-based model to predict appropriate solvents, and a list of potential solvents and solvent systems for preliminary feasibility testing. Results from Task 3.0 include experimental evaluation of epoxy removal with solvents identified in Tasks 1.0 and 2.0.

### **2.1 Information from AGMC**

#### **2.1.1 Key Criteria for Development of a New Epoxy Removal Process**

Key criteria to be met in developing an alternate epoxy removal process include:

- **Environmental Compatibility** -- The new process should be more environmentally compatible than currently used processes. Biodegradable solvents would be preferred, if available.
- **Mechanical Compatibility** -- The new process should not damage the part, either by changing dimensions or surface finish.
- **Safety** -- The new process should be safe for use by AGMC personnel in the existing repair areas.

- **Efficiency, Cost Effectiveness** -- The new process should not require more time or labor than current processes, and the cost, including labor and materials, should be comparable or less than that of current processes.

### **2.1.2 Candidate Parts for Epoxy Removal at AGMC**

Candidate parts for epoxy removal range from small, relatively simple parts such as rings or screws, with a typical dimension of about 0.2 inch made of a single metal, to complex sub-assemblies, with a typical dimension of about 5 inches, made of several different metals, plastic or other insulating components, and, possibly, several different epoxy adhesives or coatings. The parts from which epoxy is removed have lifetimes of anywhere from one day to twenty years at 125 to 170 F for a missile system, and one day to a year (on average) for an aircraft. The parts are only removed when there is a malfunction.

Typical substrate materials in these parts may include: aluminum alloys, beryllium, nickel alloys, iron, stainless steel, copper, brass, CRES, and diallyl plastic. Typical epoxies to be removed from the parts include: fluid epoxies such as FA8/BA5, LCA9/BA5, or Epon 828 with Versamid 125 or Versamid 115 activator; nonmagnetic epoxies such as LCA4XM/BA5XM; low thermal coefficient epoxies such as LCA4/BA5, LCA21/BA41, or Adhesive C-7 with Activator W; or thermally conductive epoxies such as Eccobond 2216.

### **2.1.3 Current Processes Used for Removal of Epoxy from AGMC Parts**

Epoxy currently is removed from parts by three general processes: immersion in a solvent or solvent mixture, localized application of a solvent, and/or mechanical removal of the epoxy. Immersion processes include a DMSO/nitric acid bath, a methylene chloride soak in a pressure reactor, and a soak in acetone or freon. Some of the immersion processes may involve some mechanical scraping to completely remove the epoxy.

## **2.2 Literature Search**

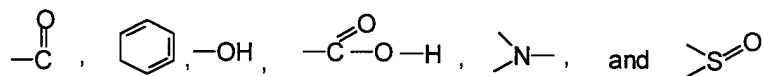
A focused literature search was carried out to identify possible candidate solvents. The search was divided into two portions. The first dealt with the available books,

conference proceedings, and product literature dealing with solvent substitution methods, biodegradable solvents, and epoxy information. A bibliography was assembled and the information was collected in a draft summary which is given in Appendix A. Producers of commercial epoxy removal solvents mentioned in conference proceedings were contacted for further information and product literature.

The second part of the literature search consisted of an on-line Chemical Abstracts literature search using the keywords: "epoxy or epoxies", "solvent", and "removal". This resulted in 112 citations. After examining the abstracts and keywords, the pertinent citations were categorized by method used, i.e., solvent removal, laser ablation removal, bead blasting removal etc. A number of citations were ordered. Table 1 summarizes the range of potential solvents identified in the literature search.

### 2.3 Solubility Parameter Modeling

The candidate solvents were examined with respect to the difference in their total solubility parameters and that of the representative epoxy, and the difference in the hydrogen bonding component of the solubility parameter. This was followed by an examination of their environmental compatibility, flash point, boiling point, and availability. A number of solvents with epoxy removal ability were found during the course of the literature search. These were examined using the solvent selection model described more fully in Appendix B. The results are listed in Table 2 along with the solubility parameters and the specific interactions. The results confirmed our earlier belief that a good solvent for epoxy removal must have solubility parameters,  $\delta^t$  and  $\delta^h$ , close to the solubility parameters of the epoxy. Smaller molar volume and polar groups--such as



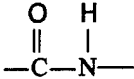





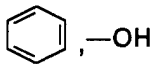
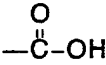
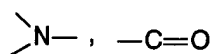
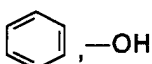
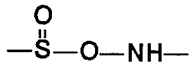

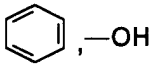
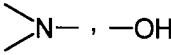
will also help by enhancing the rate of diffusion of the solvent and by providing strong interaction with epoxy adhesive.

TABLE 1. CANDIDATE SOLVENT SYSTEMS LOCATED IN LITERATURE SEARCH

Solvent System	Application	Comment	Reference
Ten percent acetone in N-methyl pyrrolidone (NMP) or ten percent methanol in anisole.	Coating removal.	Swelling is faster with mixed reagents. Need to consider in other combinations to be evaluated.	1
Actrel and Exxate cleaners.	Alternative commercial cleaners.	Proprietary compositions should be tested.	2
Hydrocarbon oils: 45 to 55 volume percent paraffin hydrocarbon and 30 to 45 volume percent naphthenic hydrocarbon.	Solvent for removing adhesive.	Need to test typical composition to determine applicability.	3
Possible useful reagents include lactic acid, ethanol, methanol, acetic acid, acetone, citric acid, toluene, oleic acid, ethyl acetate, sodium carbonate, sodium hydroxide, hydrogen peroxide, and water.	Resistance of epoxy resin at room temperature to 31 chemicals.	May be able to extract useful information from 88-week study to guide selection of environmentally benign materials.	4
NMP, ethanalamine, non-ionic surfactant.	Sprayable hot stripping agent.	Sprayable at 170 to 190 F. Developed by Navy as coating remover.	5
Methylene chloride containing glycols and cresylic and/or formic acid.	Stripping reagent for baked on coatings.	Likely to be corrosive to some metal parts. Contains methylene chloride.	6
Diethylene glycol and phosphoric acid.	Cleaning lithographic plates.	May be corrosive to some parts.	7
Cyclic alcohols with boiling point > 110 C containing small amounts of surfactant or other alcohols or ketones with boiling point > 100 C.	Integrated circuit modules.	Benzyl alcohol has been used to soften coatings on airplanes. Should evaluate applicability of range of candidates meeting these criteria.	8

(a) See References on page 26.

TABLE 2. RESULTS OF SOLUBILITY PARAMETER ANALYSIS

Solvent	Molar Volume, ml	Solubility Parameters, MPa <sup>-1/2</sup>				Interacting Groups
		$\delta^d$	$\delta^p$	$\delta^h$	$\delta^t$	
Dimethyl Formamide	77.0	17.4	34.7	11.3	24.8	
Acetone	74.0	15.5	10.4	7.0	20.0	
Methylene Chloride	63.9	28.2	6.3	6.1	20.3	
Methanol	40.7	15.1	12.3	22.3	29.6	
Toluene	106.8	18.0	1.4	2.0	18.2	
Dimethyl Sulfoxide	71.3	18.4	16.4	10.2	26.7	
Phenol	87.5	18.0	5.9	14.9	24.1	
Acetic acid	57.1	14.5	8.0	13.5	24.9	
NMP	96.5	18.0	12.3	7.2	22.9	
Benzyl Alcohol	103.6	18.4	6.3	13.7	23.8	
N-Methyl Sulfonamide	71.3	18.4	16.4	10.2	26.7	
Cyclohexanone	104.0	17.8	6.3	5.1	19.0	
Catechol (Resorcinol)	87.5	18.0	8.4	21.1	29.0	
Triethanolamine	60.2	17.2	15.6	21.3	31.5	

Based on this study, we selected five neat solvents and four mixtures for swelling/cracking tests. The solubility parameter of the mixtures was calculated by assuming that the contribution of each solvent towards  $\delta$  is proportional to its volume fraction. Thus,  $\delta_{12} = \delta_1\phi_1 + \delta_2\phi_2$ , where  $\delta_{12}$  = solubility parameter of the mixture,  $\delta_1$ ,  $\phi_1$  and  $\delta_2$ ,  $\phi_2$  are the solubility parameter and volume fraction of the two neat solvents.

## **2.4 Summary of Candidate Solvent Systems and Epoxies**

In Tasks 1.0 and 2.0 an initial group of fourteen candidate solvent systems, including neat solvents, mixtures of solvents, and commercially available compositions was defined. These solvent systems are shown in Tables 3 and 4.

One key factor in narrowing the list of candidate solvents was the possibility of hydrogen embrittlement of ferrous alloys caused by the solvent. A recent study<sup>(9)</sup> of the hydrogen embrittlement potential of eighteen formulations of paint strippers showed that ethanolamine and formic acid caused hydrogen embrittlement. Ethanolamine was a common component of the commercial formulations obtained for this epoxy removal study. Because of the potential for embrittlement, most of the mixtures containing ethanolamine were eliminated from the list of possible candidates.

A representative group of epoxies used by AGMC was chosen for testing. Formulations and their suppliers are shown in Table 5.

## **2.5 Summary of Results from Preliminary Feasibility Experiments**

These tests were performed to evaluate the potential of selected solvents and solvent systems for swelling, softening, and removing epoxy adhesives from typical component parts repaired by AGMC.

### **2.5.1 Summary of Results from Swelling/Cracking Tests**

Swelling/cracking tests were carried out for one representative epoxy composition, LCA4/BA5, and the fourteen solvents. Results from these tests showed that the three most promising solvents were N-methyl pyrrolidone (NMP), benzyl alcohol and furfuryl alcohol.



TABLE 3. SELECTED SOLVENTS AND SOLVENT MIXTURES

Type	No.	Solvent	Molar Volume, ml	Solubility Parameters, MPa <sup>-1/2</sup>		
				$\delta^d$	$\delta^p$	$\delta^t$
Neat	1	N-Methyl Pyrrolidone	96.5	18.0	12.3	7.2
	2	Benzyl Alcohol	103.6	18.0	8.2	13.7
	3	Acetophenone	117.4	19.6	8.6	3.7
	4	Ethyl Lactate	115.0	16.0	7.6	12.5
	5	Furfuryl Alcohol	86.0	17.4	7.6	15.1
Mixtures	6	Benzyl Alcohol/N-Methyl Pyrrolidone (50/50)	100.0	18.0	10.2	10.5
	7	N-Methyl Pyrrolidone/Furfuryl Alcohol (70/30)	93.4	17.8	10.9	9.8
	8	2 Pyrrolidone/Cyclohexanol (50/50)	91.2	18.4	10.3	12.4
	9	Anisole/Ethanol with 10% Sodium Ethoxide (50/50)	88.9	16.8	6.3	9.7

TABLE 4. SELECTED COMMERCIALY AVAILABLE SOLVENTS

Type	No.	Product Name	Supplier
Commercial	10	Exxate 700	Exxon Chemical Company
	11	Oakite Stripper A2	Oakite Products Company
	12	Oakite Stripper ALM	Oakite Products Company
	13	BioT Max	BioChem Systems
	14	CEE BEE A-245	McGean - Rohco, Inc.

TABLE 5. EPOXY TEST CANDIDATES

Epoxy	Supplier
FA8/BA5	Bacon Industries, Inc.
LCA9/BA5	Bacon Industries, Inc.
LCA4/BA5	Bacon Industries, Inc.
LCA4XM/BA5XM	Bacon Industries, Inc.
Adhesive C-7/Activator W	Morton International, Inc.
Epon 828/Versamid 125	Miller-Stephenson Chemical Co.
Eccobond 2216	3M, Inc.

### **2.5.2 Summary of Results from Epoxy Removal Tests**

**Neat Solvents.** Epoxy removal tests were performed for these three promising solvents with aluminum coupons coated with the seven epoxy systems. Coupons were exposed to neat solvents for six hours at 70 C and for twenty-four hours at 100 C. Two of the adhesives were removed by using neat solvent, and a third was softened substantially. Benzyl alcohol and furfuryl alcohol were more effective than N-methyl pyrrolidone for the conditions tested. The remaining epoxies were largely unaffected unless there was a chip or crack in the epoxy surface.

The chemical makeup of the seven epoxies was reviewed to determine why some of the adhesives were susceptible to the test solvents and others were unaffected. The review showed that the unaffected epoxies were highly crosslinked, which reduces the swelling and diffusion of the solvents into the epoxy, while those that were removed by the test solvents were loosely crosslinked. Addition of water or an acid catalyst to the solvent was proposed as a means to break some of the crosslinks and aid penetration of the solvent into the epoxy. Tests were carried out with mixtures of solvent and water or acid to test this hypothesis.

**Solvent Mixtures.** Cured epoxy-coated aluminum coupons were exposed to mixtures of the three solvents with 15 percent water for twenty-four hours at 70 C. Performance of all three solvents was enhanced substantially by the addition of water, as suggested above. All the tested epoxies were removed easily after exposure to one or more of the solvent mixtures

Mixtures of solvent with acid were also tested to further confirm the effect of breaking the crosslinks on epoxy removal. Epoxy removal results were generally similar to the results obtained with solvent/water mixtures.

**AGMC Parts.** Six different types of sample parts supplied by AGMC were exposed to solvent/water mixtures for twenty-four hours at 70 C. These tests confirmed that the epoxy was either completely removed by soaking or that mechanical removal was made much easier.

### **2.5.3 Summary of Results of Beryllium Exposure Tests**

Beryllium coupons supplied by AGMC were exposed to the solvent/water mixtures for approximately forty-one hours at room temperature and 70 C. It appears that no interaction between the beryllium and the solvent mixtures occurred during this exposure.

## **2.6 Detailed Results of Preliminary Feasibility Experiments**

Swelling/cracking tests were carried out with cured bulk epoxy coupons to provide initial understanding of the effect of candidate solvents on typical cured epoxies, and allowed the initial group of fourteen candidate solvent systems to be reduced to three. Epoxy removal tests were carried out with cured, epoxy-coated aluminum coupons to demonstrate the removal of cured epoxy from metal surfaces. Epoxy removal tests also were carried out with parts provided by AGMC to demonstrate the removal of epoxy from metals in the most realistic situation possible.

AGMC supplied cured bulk epoxy samples and cured epoxy-coated, aluminum lap shear test coupons for seven different epoxy formulations available from a previous study, "Experimental Evaluation of the Adhesive Degradation Potential of Aqueous Cleaning Processes".<sup>(10)</sup>

### **2.6.1 Swelling/Cracking Tests with Cured Bulk Epoxy Samples**

**Procedure.** One of the common epoxies in use at AGMC, LCA4/BA5, was chosen as the representative epoxy for the swelling/cracking tests. Small coupons (approximately 1 inch x 0.25 inch x 0.1 inch) were cut from cured bulk epoxy. The cut pieces were rinsed with methanol and dried in an oven at 70 C for one hour. Five neat solvents, four solvent mixtures and five commercial mixtures were used for the swelling study. Three coupons were used for each solvent, and for each time increment, giving a total of nine coupons for each solvent. The time increments were one hour, six hours, and twenty-four hours. Each cleaned piece was weighed and placed in a small glass vial. Solvent (25 ml) was added and the vial was sealed using a Teflon® liner. It then was placed in an oven at 70 C for the

specified length of time. After the appropriate time, the coupons were removed, blotted dry with a microwipe and weighed.

**Results.** Results from the swelling/cracking tests, presented in Table 6, show that the three most promising solvents are N-methyl pyrrolidone (NMP), benzyl alcohol and furfuryl alcohol. Weight changes for the NMP trials were impossible to determine since the solvent degraded the epoxy so much that weight change due to swelling was not a meaningful measurement. Slight degradation was also seen with benzyl alcohol and furfuryl alcohol. None of the other test solvents or commercial mixtures exhibited good results with either swelling or with epoxy degradation. The commercial products were primarily developed as cleaners and paint strippers, and may not have been intended to remove cured epoxies.

#### **2.6.2 Epoxy Removal Tests with Neat Solvents and Cured, Epoxy-Coated Metal Coupons**

**Procedure.** Epoxy removal tests were carried out for N-methyl pyrrolidone, furfuryl alcohol and benzyl alcohol, using aluminum coupons coated with the seven epoxy systems shown in Table 5. Three replicates were run for each solvent system. Coupons of each type were examined and notations were made about the epoxy coating, for example, thickness, roughness of epoxy coating, or any exposed metal. The coupons were soaked in the selected solvents for a set time, at a set temperature, placed in an ultrasonic bath for five minutes, wiped with a microwipe, and, if necessary, scraped with a Teflon spatula or an X-acto® knife. Any solid present in the solvent was noted, as well as whether the epoxy could be wiped off with a microwipe or scraped off with the spatula or knife. For each type of epoxy, the solvents were rated on performance -- ease of removal, softness of epoxy, appearance of clean metal, etc.

**Results.** Results of tests with neat solvents at six hours and 70 C are shown in Table 7, and results from tests of selected epoxies with neat solvents at twenty-four hours and 100 C are shown in Table 8.

TABLE 6. RESULTS OF SWELLING/CRACKING TESTS

Solvent	Time, hrs	Average Percent Weight Change	Comments
N-methyl pyrrolidone	1	1.10	White "scales" on surface.
	6	(a)	Deteriorating, destroyed surface.
	24	(a)	Epoxy falling apart, totally degraded.
Benzyl Alcohol	1	0.49	No change in appearance.
	6	1.51	No change in appearance.
	24	6.07	Edges swollen, waxy, crumbling slightly.
Acetophenone	1	0.44	No change in appearance.
	6	0.77	No change in appearance.
	24	2.85	One of the three pieces has yellowed.
Ethyl Lactate	1	0.27	No change in appearance.
	6	1.20	No change in appearance.
	24	2.47	Bleached.
Furfuryl Alcohol	1	1.56	Yellow (due to solvent).
	6	5.81	Darker, brittle edges.
	24	7.19	Broken edges, crumbling.
Benzyl Alcohol/N-methyl pyrrolidone (50/50 mixture)	1	0.51	No change in appearance.
	6	2.21	No change in appearance.
	24	4.28	Slightly yellowed.
N-methyl pyrrolidone/ Furfuryl alcohol (70/30)	1	0.81	Yellow (from alcohol).
	6	3.65	Brittle edges, yellow.
	24	6.31	Slightly crumbling.
2-Pyrrolidone/ Cyclohexanol (50/50)	1	0.45	No change in appearance.
	6	1.08	No change in appearance.
	24	0.92	No change in appearance.
Anisole/10% Sodium Ethoxide in Ethanol	1	0.56	surface bleached slightly
	6	3.52	bleached
	24	5.51	bleached
Exxate 700	1	0.00	no change in appearance
	6	0.00	no change in appearance
	24	0.18	no change in appearance

TABLE 6. RESULTS OF SWELLING/CRACKING TESTS (Continued)

Solvent	Time, hrs	Average Percent Weight Change	Comments
Oakite Stripper A2	1	0.53	no change in appearance
	6	0.72	no change in appearance
	24	1.43	no change in appearance
Oakite Stripper ALM	1	0.50 <sup>(b)</sup>	no change in appearance
	6	0.73	no change in appearance
	24	1.98	no change in appearance
BioT Max	1	0.00	no change in appearance
	6	0.03	no change in appearance
	24	0.13	no change in appearance
CEE-BEE A-245	1	0.29	no change in appearance
	6	0.53	no change in appearance
	24	0.73	no change in appearance

(a) Not applicable.

(b) Only one sample.

TABLE 7. RESULTS OF EPOXY REMOVAL TESTS - NEAT SOLVENTS (SIX HOURS AT 70 C)

Epoxy	N-Methyl Pyrrolidone	Benzyl Alcohol	Furfuryl Alcohol
<b>FA8/BA5</b>	Solid in solvent. Surface of epoxy was rough. Teflon spatula did not scratch surface.	Surface had degraded. Some fairly clean spots. Could chip off some epoxy with spatula.	Solid in solvent. Could chip off some epoxy. Lower layer could not be removed.
<b>LCA9/BA5</b>	No degradation of epoxy. A small amount was chipped off.	No degradation of epoxy. Spatula did not scratch surface.	No degradation of epoxy. Epoxy was quite hard. Epoxy over air bubble lifted off easily exposing clean metal.
<b>LCA4/BA5</b>	Slightly degraded. Solid in solvent. Could scrape off top layer. Epoxy was waxy.	Surface was rough. Could scrape off small amount of top layer.	Small amount of solid in solvent. Could chip off a small amount along edges of coupon.
<b>LCA4XM/ BA5XM</b>	Solid in solvent. Could rub top layer off with microwipe and could scrape off more epoxy with spatula.	No degradation of epoxy. Still quite hard.	Small amount of solid in solvent. Could wipe off some epoxy. Spatula did not scratch it.
<b>Adhesive C-7/ Activator W</b>	Solid in solvent. Could wipe off top layer. Could scrape some off with spatula.	Solid in solvent. Could wipe off top layer.	Large amount of solid in solvent. Could wipe off a good amount. Parts of coupon were totally clean.
<b>Epon 828/ Versamid 125</b>	Solid in solvent. Some could be wiped off. Spatula could chip off a bit.	Small amount of epoxy in solvent. Could wipe coupon almost clean.	Large amount of solid in solvent. One coupon was almost totally clean. Can scrape off with spatula.
<b>Eccobond 2216</b>	Solid in solvent. Spatula barely scratches surface.	Could wipe off a good part of epoxy on one coupon. Spatula could scrape surface.	One coupon wiped almost totally clean. Spatula could scrape down to metal.



TABLE 8. RESULTS OF EPOXY REMOVAL TESTS - NEAT SOLVENTS (24 HOURS AT 100 C)

Epoxy	N-Methyl Pyrrolidone	Benzyl Alcohol	Furfuryl Alcohol
<b>FA8/BA5</b>	Still had roughened, yellowed epoxy remaining. Could wipe off a small amount of material. Spatula did not scratch epoxy off. Scissors could easily scrape down to metal. Epoxy is "scaly".	Small amount of top surface wiped off. Still tough surface - was not roughened like it was with NMP. Spatula did not scratch surface.	Only a small amount could be wiped off. Epoxy is still solid.
	<b>LCA9/BA5</b>	Worked from exposed metal. Spatula was able to chip a tiny amount off. Scissors did not scratch surface of epoxy.	Spatula did not chip or scratch surface. Scissors lightly scratch the very top layer of epoxy.

Table 7 shows that two of the adhesives (Epon 828/Versamid 125 and Eccobond 2216) were removed by using neat solvent, and a third (Adhesive C-7/Activator W) was softened substantially. Benzyl alcohol and furfuryl alcohol were more effective than N-methyl pyrrolidone for the conditions tested. The remaining epoxies were largely unaffected unless there was a chip or crack in the epoxy surface. In that case, the solvent entered the epoxy, and the adhesive could be removed. Penetration of the solvent into the bulk epoxy appears to play a key role.

The chemical composition of the adhesives was examined in order to provide some additional understanding of why some of the epoxies were softened and removed, while others were unaffected. The first five adhesives in Table 5 have similar chemical compositions and behaved similarly on exposure to the solvents. The sixth adhesive (Epon 828/Versimid 125) has a similar "Part A" to the first five adhesive, however there is substantial difference in the curing agent. The curing agents (BA5 and silicic acid) in the first five epoxies have higher functionality, and form dense networks, while the curing agent in Epon 828/Versamid 125 has lower functionality, and therefore provides lower crosslink density. The seventh epoxy (Eccobond 2216) has a similar lower crosslink density. A dense network leads to smaller molecular weight between two crosslinks ( $M_c$ ), whereas a system with loose crosslinks will have higher  $M_c$ . Since the swelling coefficient,  $q$ , (see Equation 1 in Appendix B) is directly proportional to  $M_c$ , adhesives with high crosslink density will swell less than those with low crosslink density. Higher crosslink density also retards diffusion of the solvent into the epoxy.

In order to remove highly crosslinked materials, it is necessary to break down some of the crosslinks. An acid catalyst or water may be added to the solvent to hydrolyze the ether groups. The solvent mixture could then penetrate more quickly to the interface, thereby helping to split the interfacial bond and enhance delamination.

### **2.6.3 Epoxy Removal Tests with Solvent/Water Mixtures and Cured, Epoxy-Coated Metal Coupons**

**Procedure.** The procedure was similar to that used with neat solvents above. Coupons coated with the seven epoxies were exposed for twenty-four hours at 70 C to

mixtures of solvent with 15 percent water. Test solvents were N-methyl pyrrolidone, benzyl alcohol and furfuryl alcohol. For each epoxy, two coupons were examined and any special features noted. After twenty-four hours exposure to the solvent mixture, the samples were placed in the ultrasonic bath for five minutes and then the coupons were removed and examined. The amount of loose epoxy in solvent was noted as well as ease of removal with microwipe or X-acto knife.

**Results.** Results of tests with solvent/water mixtures at twenty-four hours and 70 C are shown in Table 9. Performance of all three solvents was enhanced substantially by the addition of water, as suggested in Section 2.5.2 above. All the tested epoxies were easily removed by one or more of the solvent mixtures after twenty-four hour exposure at 70 C. Mixtures with furfuryl alcohol or benzyl alcohol appear to be slightly more effective than NMP for the selected group of epoxies.

#### **2.6.4 Epoxy Removal Tests with Other Solvent Mixtures and Cured, Epoxy-Coated Metal Coupons**

In order to further test the hypothesis that an acidic solution would facilitate epoxy removal, mixtures of the three solvents containing two different acids, p-toluene sulfonic acid and glacial acetic acid, were tested on one epoxy (LCA4/BA5).

**Procedure.** P-toluene sulfonic acid (10 ml of 15 percent solution in water) was added to 90 ml each of N-methyl pyrrolidone, benzyl alcohol and furfuryl alcohol. Acetic acid (5 ml) was added to 95 ml of each solvent. Coupons coated with LCA4/BA5 were immersed in the solvent/acid mixtures for twenty four hours at 70 C followed by immersion in an ultrasound bath for five minutes. The coupons were removed, dried and examined for ease of epoxy removal.

**Results.** Results from these solvent/acid tests are shown in Table 10. NMP and benzyl alcohol gave positive results with p-toluene sulfonic acid and water, but furfuryl alcohol polymerized with the acid. Epoxy removal results were generally similar to the results obtained with solvent/water mixtures. However, in at least one case the acid appeared to interact with the substrate metal. Solvent mixtures with water and 1.5 percent

TABLE 9. RESULTS OF EPOXY REMOVAL TESTS - SOLVENT/WATER MIXTURES (24 HOURS AT 70 C)

Epoxy	N-Methyl Pyrrolidone	Benzyl Alcohol	Furfuryl Alcohol
<b>FA8/BA5</b>	Could wipe off a good deal of the epoxy. Solid in solvent. X-acto knife could scrape off remaining epoxy.	Metal almost totally clean without wiping. X-acto knife easily scraped off remaining epoxy. Very clean coupons.	Solid in solvent. One coupon wiped totally clean. The other had small amount of epoxy left which was easily taken off with X-acto knife.
<b>LCA9/BA5</b>	Loose epoxy above air bubble. No noticeable amount could be wiped off. With effort, epoxy could be removed with X-acto knife.	Loose epoxy over air bubble. Took effort to remove with X-acto knife.	Solid in solvent. Coupon wiped totally clean. Half of other coupon was wiped clean. X-acto knife could peel off remaining epoxy.
<b>LCA4/BA5</b>	No solid seen in solvent. Top layer of epoxy could be wiped off. Still hard underneath. Used X-acto knife to scrape off remaining epoxy with little effort.	Small amount of solid in solvent. More epoxy could be wiped off (compared to NMP). Still some solid epoxy present but X-acto knife took it off.	Half of epoxy was removed on one coupon. Very little epoxy wiped off. Difficult to scrape off epoxy with X-acto knife.
<b>LCA4XM/ BA5XM</b>	Solid in solvent. Could wipe off top layer of epoxy. Fairly difficult to scrape off with X-acto knife.	Solid in solvent. Top layer wiped off. Can scrape off layers with X-acto knife but was not easy to get down to clean metal.	Solid in solvent. Epoxy peeled off one corner of one coupon. Top layer wiped off. Could scrape down to metal with some effort using X-acto knife.
<b>Adhesive C-7/ Activator W</b>	No solid in solvent. Could wipe off some "scales". Easy to scrape off with X-acto knife.	Solid in solvent. Could wipe coupons almost totally clean. Only a small amount of scraping needed with X-acto knife.	Coupons almost totally clean when removed from solvent. Tiny specks remained which were easily taken off with X-acto knife.
<b>Epon 828/ Versamid 125</b>	Solid in solvent. Both coupons wiped almost totally clean. Easy to scrape off remaining epoxy.	Both coupons clean when taken out of solvent.	Solid in solvent. One coupon was almost completely clean. Both coupons were clean when wiped with a microwipe.
<b>Eccobond 2216</b>	Some solid in solvent. Top layer of epoxy was wiped off. X-acto knife could easily scrape off remaining epoxy.	Epoxy peeled off both coupons in one solid strip. Clean metal when taken out of solvent.	Both coupons came out clean.

Note: Shading indicates the most effective solvent mixture or mixtures for each epoxy.

TABLE 10. RESULTS OF EPOXY REMOVAL TESTS - SOLVENT/ACID MIXTURES (24 HOURS AT 70 C)

Acid	N-Methyl Pyrrolidone	Benzyl Alcohol	Furfuryl Alcohol
1.5 percent P-toluene sulfonic acid and water	Neutral with indicator paper. Could wipe off thinner coat easily. Exposed clean metal. Thicker coat on other coupon had almost completely rubbed off. Epoxy was soft - easy to scrape off.	Solvent was dark grey. Acidic. Aluminum was discolored. Thinner epoxy wiped off exposing clean metal. Remaining epoxy was still tough but X-acto knife did peel it off.	Solvent had been polymerized. Impossible to examine coupons.
Glacial acetic acid	Neutral. Solid in solvent. Good deal of top layers were wiped off but not down to clean metal. Epoxy was still tough, especially the layers closest to the metal surface.	Acidic. Solid in solvent. One coupon wiped down to a thin layer of epoxy. Could scrape epoxy off with knife but was still tough.	Acidic. Solid in solvent. One coupon wiped clean except for a few "dots" of epoxy which were easily scraped off. Other coupon had some metal exposed but the epoxy was tough and quite hard to scrape off.

P-toluene sulfonic acid appeared to remove the epoxy more easily and completely than glacial acetic acid.

#### **2.6.5 Epoxy Removal Tests with AGMC-Supplied Parts**

**Procedure.** Three examples of each of six different sample parts were provided by AGMC. Each part was carefully examined using a magnifier. Any imperfections in the metal were noted as well as location of epoxy, appearance of metal etc. The three parts, depending upon the size of the part, were placed in a glass or polyethylene container, and a solvent/water mixture was added to the container (specific solvent is noted in Table 11). After exposure at 70 C for twenty-four hours, the containers were placed in the ultrasound bath for five minutes. The parts were removed from the container and epoxy was wiped or scraped from the parts as necessary. The parts were then dried and carefully examined with a magnifier.

**Results.** Results of tests with AGMC-supplied parts are presented in Table 11. Epoxy removal results were very positive. These tests confirmed that the specified solvent/water mixtures either removed the epoxy or made mechanical removal easier. However, furfuryl alcohol/water appeared to interact in some way with a bearing and shaft assembly. This assembly included a bearing which may have contained residual lubricant which could have been dissolved by the solvent mixture. This might result in the discoloration of the polished chromium alloy surface noted in the table. Further testing would be necessary to confirm that the specific solvent-water mixture does not interact unfavorably with substrate metals.

The results of this testing showed even more complete removal of the epoxies than the tests with aluminum coupons. This may be because the epoxy on the actual parts is in thinner layers, or because the adhesive on the coupons was cured more thoroughly. It may also be due to smoother metal surfaces on the actual parts compared to the coupons, which were etched prior to bonding with the adhesive.

TABLE 11. RESULTS OF PARTS TESTING (24 HOURS AT 70 C)

Part	Part Composition	Solvent Mixture	Comments
Seal Ring Cover	Mumetal with LCA4/BA5 epoxy staking along outer side of part	Benzyl Alcohol/Water	All the epoxy staking was removed by the solvent. No scraping necessary. Dried parts looked completely clean. Metal was not damaged.
Shunt	Ni-Fe with EA934NA or EponIV/ Curing Agent A	Furfuryl Alcohol/Water	Pieces of loose epoxy in solvent. Dried with microwipe. Part was very clean, no epoxy remaining, metal was clean, shiny, and undamaged.
Bearing and Shaft Assembly	Chromium alloy with FA8/BA5	Furfuryl Alcohol/Water	Solvent was dark brown and opaque. The two outer sections of part that had epoxy on them were discolored. The metal was darker and duller in spots, while the middle section appeared clean and shiny. These darker sections seem to be where the epoxy had coated the metal. All the epoxy appeared to have been removed by the solvent. The shaft of the part was also much darker and a small amount of black, soot-like material was removed.
Magnet Sub-Assembly	Nickel-iron based alloy with LCA4/BA5	Furfuryl Alcohol/Water	Loose epoxy was seen in solvent. Some of the more accessible staking wiped right off. However, in the tighter crevices, the epoxy had to be picked out using a narrow X-acto knife. The worst case was trying to remove the epoxy from a 90 degree corner. More epoxy was more easily removed from two of the three pieces. No idea why the third piece didn't respond as well since they all had similar exposure to the solvent.
Retaining Bearing Ring	CRES with LCA4XM/BA5XM	Furfuryl Alcohol/Water	Loose epoxy in solvent. No epoxy remained on parts. Metal appeared undamaged.
Tooth Spur Gear	Ni-Cr steel with LCA4XM/BA5XM	Benzyl Alcohol/Water	Strands of epoxy in solvent. Parts dried with microwipe. Metal was clean and shiny. A very few specks of epoxy were seen (2-3 per part) and were easily removed by wiping or by picking gently with X-acto knife.

### **2.6.6 Beryllium Exposure to Solvent/Water Mixtures**

**Procedure.** AGMC supplied beryllium coupons for preliminary evaluation of potential interaction with the three solvent/water mixtures. The coupons were rinsed with distilled water and acetone. When dried, the coupons were weighed and the general appearance was noted. Each coupon was placed in one of the three test mixtures solvents (N-methyl pyrrolidone, benzyl alcohol or furfuryl alcohol) with 15 percent water added. The jars were sealed using Teflon liners. After exposure for one hour at room temperature, observations were recorded and the jars were left undisturbed for a further sixteen hours. Again, observations were recorded. The test containers were then heated to 70 C and held there for eight hours. Observations were recorded at several points. Exposure was then continued for sixteen hours at room temperature. At this point, the coupons were removed, washed well with acetone, weighed and examined for any surface changes.

**Results.** Initially, the coupon used with NMP was slightly discolored on one side with darker spots on the surface which may have been the result of machining. The other two coupons appeared clean and smooth. Once the solvent was added bubbles appeared on the top and bottom surfaces of the coupons, especially the NMP coupon. After sitting for sixteen hours at room temperature, more bubbles appeared on the NMP coupon, fewer on the benzyl alcohol coupon, and fewer still on the furfuryl alcohol coupon. When heated, no bubbles were present on the NMP sample and the furfuryl alcohol sample with only a few small ones on the benzyl alcohol sample. After sitting once again for sixteen hours at room temperature, there were only a very few small bubbles on any of the samples. The coupons showed no weight loss during exposure. It is likely that the bubbles that formed on the surface were composed of gas dissolved in the solvent mixture, nucleating on the slightly rough surface of the coupons.

At the conclusion of the exposure, the NMP coupon still showed some darker spots on the surface. The benzyl alcohol coupon showed some random small, dark dots on the surface, and the furfuryl alcohol coupon showed a few darker spots on one side. More detailed study is recommended to confirm that these solvent/water mixtures do not affect beryllium surfaces typical of parts repaired by AGMC.



### **3.0 CONCLUSIONS**

The objective of this first phase program was to identify alternative, solvent-based methods for epoxy removal which are efficient, more environmentally compatible than current processes, safe in their application, and which do not degrade the reliability of the equipment being repaired and maintained by AGMC. The results of this phase of the program, including the preliminary feasibility experiments, provide the following conclusions:

1. Three promising solvents for use in development of an alternative, solvent-based method for epoxy removal have been identified:
  - N-methyl pyrrolidone
  - Benzyl alcohol
  - Furfuryl alcohol
2. Mixtures of these solvents with water gave much better results for epoxy removal than neat solvents.
3. Exposure time of twenty-four hours at 70 C was sufficient to soften or remove the seven epoxies used in these tests. It may be possible to use shorter exposure times and/or lower temperatures, but that should be verified by further testing.
4. These three solvents have been identified as environmentally compatible, and safe for use in appropriately ventilated work facilities. Material Safety Data Sheets for the three solvents are included in Appendix C.
5. The three solvent-water mixtures appear to be generally compatible with metal substrates common to AGMC parts, particularly with beryllium. However these tests were preliminary and more complete testing would be necessary to confirm compatibility.

### **4.0 RECOMMENDATIONS**

The results of the first phase of the program are sufficiently promising to recommend that a second phase of the program be carried out. This second phase program should focus

on developing an epoxy removal process based on the use of the candidate solvents identified above. Work in the second phase should include:

1. Comparison of the candidate solvents
2. Enhancement of the performance of the selected solvents through additives such as surfactants or corrosion inhibitors
3. Evaluation of the potential for damage to metal substrates typical of parts repaired by AGMC
4. Optimization of one or more cleaning processes for removing cured epoxy adhesives or coatings from typical parts repaired by AGMC.

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**APPENDIX A**

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## **APPENDIX B**

### **IDENTIFICATION OF SOLVENTS FOR EPOXY REMOVAL** **BY SOLUBILITY PARAMETER MODEL**

# IDENTIFICATION OF SOLVENTS FOR EPOXY REMOVAL BY SOLUBILITY PARAMETER MODEL

## INTRODUCTION

One approach to removing epoxy adhesives is to swell them in a solvent and remove the swollen gel by mechanical means. The swelling of the crosslinked network is governed by two forces:

1. The osmotic force which is proportional to the free energy of mixing ( $\Delta G_m$ ).
2. The elastic force which is proportional to the crosslink density and the elastic modulus of the network.

While the osmotic force transports the solvent into the crosslinked epoxy mass, the elastic force opposes the solvent movement. The thermodynamics of this process has been worked out by Flory & Rehner<sup>(1)</sup> assuming that the network is lightly and randomly crosslinked and the free energy of mixing is given by the Flory-Huggins equation. The swelling coefficient, which is the volume of the solvent absorbed by a unit of volume of the crosslinked network at equilibrium is given by

$$q^{5/3} \approx (\bar{v}M_c)(1 - 2M_c/M)^{-1}(0.5 - \chi)/\phi_s \quad (1)$$

where  $\bar{v}$  is specific volume,  $M_c$  is the molecular weight between crosslinks, and  $M$  is the molecular weight of the polymer,  $\chi$  is the Flory interaction parameter for the polymer/solvent system, and  $\phi_s$  is the volume fraction of the solvent. This term  $\chi$  comes from the Flory-Huggins equation for the free energy of mixing of a polymer with a solvent and is given by

$$\Delta G_m = RT(n_s \ln \phi_s + n_p \ln \phi_p + \chi n_s \phi_p) \quad (2)$$

where  $n_s$ ,  $n_p$  are the number of moles and  $\phi_s$ ,  $\phi_p$  are the volume fractions of the solvent and polymer respectively. The first two terms in this equation represent the entropy of mixing ( $\Delta S_m$ ) and the third term represents the enthalpy of mixing ( $\Delta H_m$ ). The entropy of mixing of polymers with solvent is negligibly small, due to the long chain nature of the polymer molecules. Hence, the free energy of mixing ( $\Delta G_m$ ) is controlled by  $\Delta H_m$ . For the



polymer to mix with the solvent,  $\Delta G_m$  has to be negative or zero, and that requires  $\Delta H_m$  to be zero or very small. In other words,  $\chi$  has to be very small. Hildebrand<sup>(2)</sup> showed that for a mixing process, for which there is no volume change and the entropy change is ideal,  $\Delta H_m = KV_s(\delta_p - \delta_s)^2$  where  $\Delta H_m$  is the partial molar enthalpy of mixing,  $K$  is a constant, approximately equal to 1,  $V_s$  is the molar volume of the solvent,  $\delta_s$  and  $\delta_p$  are the solubility parameter of the solvent and the polymer respectively. In other words for  $\Delta H_m$  to be small or zero,  $\delta_s$  should approach the value of  $\delta_p$ .

The solubility parameter,  $\delta$ , is the square root of the energy of vaporization ( $\Delta E_v$ ) and is given in units of  $(\text{cal/cc})^{-1/2}$  or  $\text{MPa}^{-1/2}$ . Hence, the solubility parameter for solvents can be calculated from the enthalpy of vaporization ( $\Delta H_v$ ).  $\Delta E_v = \Delta H_v - RT$ . Alternatively, they can be calculated from molar attraction constants. In 1942, G. Gee<sup>(3)</sup> showed that Hildebrand's relation applies to polymer solutions and one can estimate  $\delta$  of polymers by measuring the relative solubility of a polymer in a series of solvents. Mangaraj<sup>(4)</sup>, et al used Gee's approach and showed that Gee's relation holds valid mainly for nonpolar systems and for systems in which the series of the solvents are of the same chemical nature as the polymer. In 1967, Hansen<sup>(5)</sup> showed that for polar polymers and solvents, Hildebrand's solubility parameter  $\delta$ , should be divided into three components,  $\delta^d$ ,  $\delta^p$ , and  $\delta^h$  representing the three constituents of intermolecular interaction, namely dispersive, dipole-dipole and hydrogen bonding interaction. Hence, for a solvent to dissolve in a polymer the individual solubility parameter components should approach each other; i.e.,

$$(\delta_s^t - \delta_p^t)^2 = (\delta_s^d - \delta_p^d)^2 + (\delta_s^p - \delta_p^p)^2 + (\delta_s^h - \delta_p^h)^2 \rightarrow 0 \quad (3)$$

$$\delta^t = (\delta_s - \delta_p) = \sqrt{(\delta_s^d - \delta_p^d)^2 + (\delta_s^p - \delta_p^p)^2 + (\delta_s^h - \delta_p^h)^2} \rightarrow 0 \quad (4)$$

The component solubility parameters of many solvents and polymers have been experimentally determined and empirical models have been suggested for estimating the same from molecular parameters<sup>(6,7)</sup>. It has been shown that for a solvent to dissolve a polymer, the critical solubility parameter difference should depend on the type of interaction<sup>(8)</sup> between the polymer and the solvent. Table B-1 presents a rough estimate of critical values of  $\Delta\delta$  for different types of interaction. Whereas the critical  $\Delta\delta_s$  is small for polymers and solvents

having weak interactions, it can be substantially larger for polymers and solvents having strong interactions. Battelle has used this approach in identifying good solvents for this program. Reference 7 gives a range of solubility parameters for epoxy compounds including their  $\delta^d$ ,  $\delta^p$ , and  $\delta^h$ . These vary within a small range depending on the type of epoxy resin, the curing agent, and the method of estimation. Selected values of these parameters are presented in Table B-2.

TABLE B-1. CRITICAL SOLUBILITY PARAMETER DIFFERENCE FOR DIFFERENT SYSTEMS

Specific Interaction	Critical $\Delta\delta$ , MPa <sup>-1/2</sup>
Dispersion Forces Alone	1 to 2.0
Moderate Polarity	2 to 4.1
Hydrogen Bonding	4 to 6.1

TABLE B-2. SOLUBILITY PARAMETER OF EPOXY RESINS, MPa<sup>-1/2</sup>

Author	$\delta^t$	$\delta^d$	$\delta^p$	$\delta^h$
Alessi, Kikic <sup>(10)</sup>	22	18	6	8.5
Hansen <sup>(9)</sup>	25.9	20.7	9.4	12.0
Hansen <sup>(5)</sup>	26.3	20.4	12.0	11.5

Initially we used the three sets of values given in Table B-2 and Battelle's data base for solvent substitution to estimate the solubility parameters differences in order to identify solvents which can dissolve or swell the epoxy resins. The candidate solvents are given in Tables B-3, B-4, and B-5 along with the root mean square solubility parameter differences ( $\Delta\delta$ ) in increasing order.

Subsequently an attempt was made to estimate the solubility parameter of the epoxy LCA4/BA5 identified by AGMC as one commonly used in their maintenance work. The two-part formulation of this epoxy is given in Table B-6.

TABLE B-3. CANDIDATE SOLVENTS BASED ON EPOXY SOLUBILITY  
PARAMETER DATA OF ALESSI AND KIKIC<sup>(10)</sup>

Solvents	$\Delta\delta^t$	$\Delta\delta^d$	$\Delta\delta^p$	$\Delta\delta^h$
Tetrahydrofuran	1.3	1.2	0.3	0.5
N-Propylamine	1.5	1.0	1.1	-0.1
Tetrachloroethane	1.5	-0.8	0.9	-0.9
Morpholine	1.5	-0.8	1.1	-0.7
Benzoic acid	1.7	-0.2	-1.0	-1.3
Quinoline	1.9	-1.4	-1.0	0.9
Aniline	2.2	-1.4	0.9	-1.5
Di-(2-methoxyethyl) ether	2.3	2.2	-0.1	-0.7
N-Butylamine	2.4	1.8	1.5	0.5
Methylene dichloride	2.4	-0.2	-0.3	2.4
Nonyl phenol	2.5	1.4	1.9	-0.7
Anisole	2.6	0.2	1.9	1.7
Dibenzyl ether	2.6	0.6	2.3	1.1
Ethanethiol	2.6	2.2	-0.6	1.3
Ethyl acetate	2.6	2.2	0.7	1.3
N-Butyl lactate	2.8	2.2	-0.5	-1.7
Isophorone	2.8	1.4	-2.2	1.1
Mesityl oxide	2.9	1.6	-0.1	2.4
Ethyl formate	2.9	2.5	-1.2	0.9
Methyl acetate	2.9	2.5	-1.2	0.9
Diethylene glycol mono-n-butylether	3.1	2.0	-1.0	-2.1
2-Ethoxyethyl acetate	3.2	2.0	1.3	-2.1
2-Octanol	3.3	1.8	1.1	-2.6
Cyclohexanone	3.4	0.2	-0.3	3.4
Cyclohexylamine	3.5	0.6	2.9	1.9
Tetramethylurea	3.6	1.2	-2.2	-2.6
1-Decanol	3.6	0.4	3.3	-1.5
Methylene diiodide	3.7	0.2	2.1	3.0
1,1-Dimethylhydrazine	3.7	2.7	0.1	-2.5
Butyraldehyde	3.7	3.3	0.7	1.5
Benzaldehyde	3.8	-1.4	-1.4	3.2
Diacetone alcohol	3.9	2.2	-2.2	-2.3

TABLE B-4. CANDIDATE SOLVENTS BASED ON EPOXY SOLUBILITY  
PARAMETER DATA OF HANSEN<sup>(9)</sup>

Solvents	$\Delta\delta^t$	$\Delta\delta^d$	$\Delta\delta^p$	$\Delta\delta^h$
Ethylene dibromide	2.9	1.1	2.6	0.4
Hexamethyl phosphonamide	2.9	-2.5	0.8	1.2
O-Methoxyphenol	3.1	2.7	1.2	-0.8
Benzyl alcohol	4.0	2.3	3.1	-1.2
Benzoic acid	4.4	2.5	2.4	2.7
Diethylene glycol monoethyl ether	4.5	4.5	0.2	0.2
Furfuryl alcohol	4.6	3.3	1.8	-2.6
Diethylene glycol monomethyl ether	4.8	4.5	1.6	-0.2
Ethylene glycol monoethyl ether	4.9	4.5	0.2	-1.8
Methyl salicylate	4.9	4.7	1.4	0.2
N,N-dimethylacetamide	5.0	3.9	-2.1	2.3
Phenol	5.0	2.7	3.5	-2.4
Ethyl lactate	5.0	4.7	1.8	0.0
M-Cresol	5.1	2.7	4.3	-0.4
Aniline	5.1	1.3	4.3	2.5
3-Chloropropanol	5.3	3.1	3.7	-2.2
Diacetone alcohol	5.3	4.9	1.2	1.7
Triethyl phosphate	5.5	3.9	-2.1	3.3
Diethylene glycol mono-n-butyl ether	5.6	4.7	2.4	1.9
Quinoline	5.6	1.3	2.4	4.9

TABLE B-5. CANDIDATE SOLVENTS BASED ON EPOXY SOLUBILITY  
PARAMETER DATA OF HANSEN<sup>(5)</sup>

Solvents	$\Delta\delta^t$	$\Delta\delta^d$	$\Delta\delta^p$	$\Delta\delta^h$
N,N-Dimethylacetamide	3.9	3.6	0.5	1.3
Triethyl phosphate	4.3	3.6	0.5	2.3
Hexamethyl phosphonamide	4.4	-2.8	3.4	0.2
Acetic anhydride	4.6	4.4	0.3	1.3
Diethylenetriamine	4.7	3.6	-3.1	-2.8
O-Methoxyphenol	4.8	2.4	3.8	-1.8
N-Methyl-2-pyrrolidone	4.9	2.4	-0.3	4.3
Dimethylsulfoxide (DMSO)	5.0	2.0	-4.4	1.3
Nonyl phenoxy ethanol	5.1	3.6	1.8	3.1
Diethylene glycol monoethyl ether	5.1	4.2	2.8	-0.8
Tetramethylurea	5.2	3.6	3.8	0.4
Ethylene dibromide	5.3	0.8	5.2	-0.6
2-Pyrrolidone	5.5	1.0	-5.4	0.2
Trimethyl phosphate	5.5	3.6	-4.0	1.3
Benzoic acid	5.7	2.2	5.0	1.7
Ethylene glycol monoethyl ether	5.8	4.2	2.8	-2.8
Methyl salicylate	6.0	4.4	4.0	-0.8
Diacetone alcohol	6.0	4.6	3.8	0.7
Diethylene glycol monomethyl ether	6.1	4.2	4.2	-1.2
Ethyl lactate	6.3	4.4	4.4	-1.0
Butyrolactone	6.3	1.4	-4.6	4.1
Quinoline	6.4	1.0	5.0	3.9

TABLE B-6. FORMULATION OF LCA4/BA5 EPOXY ADHESIVE

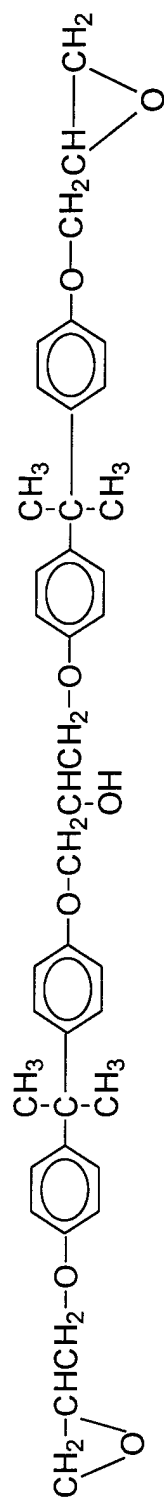
Part A		Part B	
Chemical	Percent	Chemical	Percent
4'(methylethylidene) bisphenol with chloro methyl oxirane	36	N-(amino ethyl aminoethyl)-imidazolidine	90
Limestone (calcium carbonate)	62	Silica	8
Silica	2	Calcium carbonate	<2

The organic part of this epoxy resin is the binder and has to be dissolved or swollen for adhesive removal. Figure B-1 presents the reaction between the polymeric diglyceride and the imidazolidine curing agent.

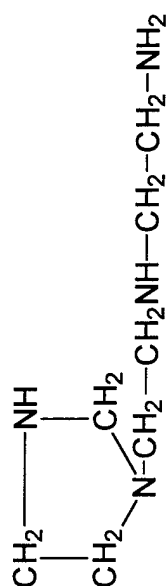
We have estimated the solubility parameter for cured epoxy LCA4/BA5, using molar attraction constants and molar volumes for each group in the structure. The molar attraction and molar volume constants were taken from "CRC Handbook on Solubility and Other Cohesion Parameters"<sup>(6)</sup>. The estimated value for  $\delta^t$  was  $23.5 \text{ MPa}^{-1/2}$ . (Without the chloromethyl side group the epoxy compound will have a solubility parameter of approximately  $22.1 \text{ MPa}^{-1/2}$ .) Since we did not have an independent estimate of  $\delta^d$ ,  $\delta^b$ , and  $\delta^h$ , they were interpolated from the three sets of component solubility parameters given in Table B-2. Results are presented in Table B-7. The average of the three sets of component solubility parameters were calculated and used for selecting candidate solvents with our computer data base. Table B-8 presents the candidate solvents.

TABLE B-7. ESTIMATED SOLUBILITY PARAMETER VALUES FOR EPOXY LCA4/BA5

	Component Solubility Parameter Values, $\text{MPa}^{-1/2}$			
	Alessi, Kikic <sup>(10)</sup>	Hansen <sup>(9)</sup>	Hansen <sup>(5)</sup>	Average
$\delta^d$	19.2	18.8	18.2	18.7
$\delta^b$	6.4	8.7	10.7	8.6
$\delta^h$	9.1	11.3	10.3	10.2



Part A



Part B

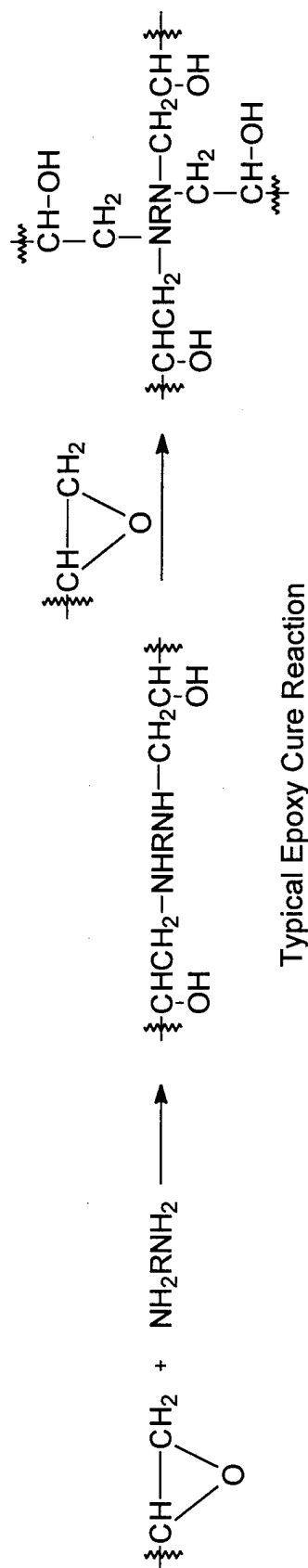


FIGURE B-1. CHEMICAL STRUCTURE OF LCA4/BA5

TABLE B-8. CANDIDATE SOLVENTS BASED ON THE ESTIMATED SOLUBILITY PARAMETER OF LCA4/BA5 EPOXY RESIN

Solvents	Solubility Parameter Difference, MPa <sup>-1/2</sup>			
	$\Delta\delta^t$	$\Delta\delta^d$	$\Delta\delta^p$	$\Delta\delta^h$
Tetramethylurea	2.1	1.9	0.4	-0.9
Ethylene dibromide	2.8	-0.9	1.8	-1.9
Diacetone alcohol	3.0	2.9	0.4	-0.6
Nonyl phenoxy ethanol	3.1	1.9	-1.6	1.8
Quinoline	3.1	-0.7	1.6	2.6
Ethylene glycol mono-n-butyl ether	3.2	2.7	1.6	-0.4
O-Methoxyphenol	3.2	0.7	0.4	-3.1
Diethylene glycol monoethyl ether	3.3	2.5	-0.6	-2.1
N,N-Dimethylacetamide	3.5	1.9	-2.9	0.0
Methyl salicylate	3.5	2.7	0.6	-2.1
Isophorone	3.5	2.1	0.4	2.8
Aniline	3.6	-0.7	3.5	0.2
N-Butyl lactate	3.6	2.9	2.1	0.0
Tetrachloroethane	3.6	-0.1	3.5	0.8
Triethyl phosphate	3.6	1.9	-2.9	1.0
Diethylene glycol monomethyl ether	3.6	2.5	0.8	-2.5
Ethyl lactate	3.7	2.7	1.0	-2.3
Morpholine	3.8	-0.1	3.7	1.0
Di-(2-methoxyethyl) ether	4.0	2.9	2.5	1.0
Tetrahydrofuran	4.1	1.9	2.9	2.2
Acetic anhydride	4.1	2.7	-3.1	0.0
Benzyl alcohol	4.2	0.3	2.3	-3.5
Pyridine	4.3	-0.3	-0.2	4.3
Ethyl formate	4.4	3.2	1.4	2.6
Methyl acetate	4.4	3.2	1.4	2.6
N-Propylamine	4.4	1.7	3.7	1.6
1,1-Dimethylhydrazine	4.4	3.4	2.7	-0.8
M-Cresol	4.5	0.7	3.5	-2.7
Bis(2-chloroethyl) ether	4.5	-0.1	-0.4	4.5
2-Octanol	4.6	2.5	3.7	-0.9
Ethanethiol	4.6	2.9	2.0	3.0
Hexamethyl phosphonamide	4.6	-4.5	0.0	-1.1
Methylene dichloride	4.7	0.5	2.3	4.1
2-Ethoxyethyl acetate	4.8	2.7	3.9	-0.4
Ethyl chloroformate	4.8	3.1	-1.4	3.4
N-Methyl-2-pyrrolidone	4.8	0.7	-3.7	3.0
Ethylene glycol monoethyl ether	4.8	2.5	-0.6	-4.1
Acetone	4.9	3.2	-1.8	3.2
Ethylene glycol mono-n-butyl ether	4.9	2.7	3.5	-2.1



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## **APPENDIX C**

### **MATERIAL SAFETY DATA SHEETS**



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**aldrich chemical co., inc.**

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 BATTELLE MEMORIAL INSTITUTE  
 505 KING AVENUE  
 COLUMBUS OH 43201-2681

 DATE: 05/07/93  
 CUST#: 130451  
 PO#: 54462

0466

## M A T E R I A L S A F E T Y D A T A S H E E T

PAGE 1

## - - - - IDENTIFICATION - - - -

 PRODUCT #: F1990-6  
 CAS #: 98-00-0  
 MF: C5H6O2

NAME: FURFURYL ALCOHOL, 98%

## SYNONYMS

 2-FURANCARBINOL \* 2-FURANMETHANOL \* FURFURAL ALCOHOL \* FURFURALCOHOL \*  
 FURFURYL ALCOHOL (ACGIH, DCT, OSHA) \* 2-FURFURYLALCOHOL (CZECH) \*  
 FURYL ALCOHOL \* ALPHA-FURYL CARBINOL \* 2-FURYL CARBINOL \* 2-  
 FURYL METHANOL \* 2-HYDROXYMETHYLFURAN \* METHANOL, (2-FURYL)- \* NCI-  
 C55224 \*

## - - - - TOXICITY HAZARDS - - - -

 RTECS #: LU9100000  
 FURFURYL ALCOHOL

## IRRITATION DATA

EYE-RBT 100 MG/24H MOD

85JCAE -,787,86

## TOXICITY DATA

 IHL-RAT LC50:233 PPM/4H  
 IPR-RAT LD50:650 MG/KG  
 SCU-RAT LD50:85 MG/KG  
 UNR-RAT LD50:460 MG/KG  
 ORL-MUS LD50:160 MG/KG  
 UNR-MUS LD50:338 MG/KG  
 SKN-RBT LD50:400 MG/KG  
 IVN-RBT LD50:650 MG/KG  
 UNR-RBT LD50:632 MG/KG

 AIHAAP 19,91,58  
 NPIRI\* 1,64,74  
 34ZIAG -,280,69  
 GISAAA 55(6),73,90  
 BIJOAK 34,1196,40  
 GISAAA 55(6),73,90  
 34ZIAG -,280,69  
 FEPR7 8,294,49  
 GISAAA 55(6),73,90

## REVIEWS, STANDARDS, AND REGULATIONS

 ACGIH TLV-TWA 10 PPM; STEL 15 PPM (SKIN) 85INA8 5,281,86  
 MSHA STANDARD-AIR: TWA 5 PPM (20 MG/M3) (SKIN) DTLWS\* 3,19,73  
 OSHA PEL: 8H TWA 50 PPM (200 MG/M3) FEREAC 54,2923,89  
 OSHA PEL FINAL: 8H TWA 10 PPM (40 MG/M3); STEL 15 PPM (60 MG/M3) (SKIN)  
 FEREAC 54,2923,89  
 OEL-AUSTRALIA: TWA 10 PPM (40 MG/M3); STEL 15 PPM (60 MG/M3); SKIN  
 JANUARY 1993  
 OEL-BELGIUM: TWA 10 PPM (40 MG/M3); STEL 15 PPM (60 MG/M3); SKIN JANUARY  
 1993  
 OEL-DENMARK: TWA 5 PPM (20 MG/M3); SKIN JANUARY 1993  
 OEL-FINLAND: TWA 5 PPM (20 MG/M3); STEL 10 PPM (40 MG/M3); SKIN JANUARY  
 1993  
 OEL-FRANCE: TWA 10 PPM (40 MG/M3) JANUARY 1993  
 OEL-GERMANY: TWA 50 PPM (200 MG/M3) JANUARY 1993  
 OEL-HUNGARY: STEL 8 MG/M3; SKIN JANUARY 1993

CONTINUED ON NEXT PAGE

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M A T E R I A L   S A F E T Y   D A T A   S H E E T

PAGE 2

CUST#: 130451  
PO#: 54462

PRODUCT #:

NAME: FURFURYL ALCOHOL, 98%

MF: C5H6O2

- - - - TOXICITY HAZARDS - - - -

OEL-JAPAN:TWA 5 PPM (20 MG/M3) JANUARY 1993  
OEL-RUSSIA:TWA 5 PPM;STEL 0.5 MG/M3;SKIN JANUARY 1993  
OEL-SWEDEN:TWA 5 PPM (20 MG/M3);STEL 10 PPM (40 MG/M3) JANUARY 1993  
OEL-SWITZERLAND:TWA 10 PPM (40 MG/M3);SKIN JANUARY 1993  
OEL-THE NETHERLANDS:TWA 5 PPM (20 MG/M3);STEL 50 PPM (200 MG/M3);SKIN  
JANUARY 1993  
OEL-THE PHILIPPINES:TWA 50 PPM (200 MG/M3) JANUARY 1993  
OEL-TURKEY:TWA 50 PPM (200 MG/M3) JANUARY 1993  
OEL-UNITED KINGDOM:TWA 5 PPM (20 MG/M3);STEL 15 PPM (60 MG/M3);SKIN  
JANUARY 1993  
OEL IN BULGARIA, COLOMBIA, JORDAN, KOREA, NEW ZEALAND, SINGAPORE,  
VIETNAM CHECK ACCIHL TLV  
NIOSH REL TO FURFURYL ALCOHOL-AIR:TWA 10 PPM (SK);STEL 15 PPM (SK)  
NIOSH# DHHS #92-100.92  
NOHS 1974: HZD 34370; NIS 29; TNF 2939; NOS 38; TNE 11577  
NOES 1983: HZD 34370; NIS 46; TNF 2827; NOS 63; TNE 77710; TFE 14624  
EPA TSCA CHEMICAL INVENTORY, JUNE 1990  
EPA TSCA TEST SUBMISSION (TSCATS) DATA BASE, JANUARY 1993  
NIOSH ANALYTICAL METHODS: SEE FURFURYL ALCOHOL 2505  
NTP CARCINOGENESIS STUDIES:ON TEST (TWO YEAR STUDIES), OCTOBER 1992

TARGET ORGAN DATA

BEHAVIORAL (CONVULSIONS OR EFFECT ON SEIZURE THRESHOLD)  
TUMORIGENIC (ACTIVE AS ANTI-CANCER AGENT)  
ONLY SELECTED REGISTRY OF TOXIC EFFECTS OF CHEMICAL SUBSTANCES  
(RTECS) DATA IS PRESENTED HERE. SEE ACTUAL ENTRY IN RTECS FOR  
COMPLETE INFORMATION.

- - - - HEALTH HAZARD DATA - - - -

ACUTE EFFECTS

HARMFUL IF SWALLOWED, INHALED, OR ABSORBED THROUGH SKIN.  
VAPOR OR MIST IS IRRITATING TO THE EYES, MUCOUS MEMBRANES AND UPPER  
RESPIRATORY TRACT.  
PROLONGED CONTACT CAN CAUSE:  
DAMAGE TO THE EYES  
CNS DEPRESSION  
DAMAGE TO THE KIDNEYS

FIRST AID

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# M A T E R I A L   S A F E T Y   D A T A   S H E E T

PAGE 3

 CUST#: 130451  
 PO#: 54462

PRODUCT #:

NAME: FURFURYL ALCOHOL, 98%

MF: C5H6O2

## - - - - HEALTH HAZARD DATA - - - -

IN CASE OF CONTACT, IMMEDIATELY FLUSH EYES OR SKIN WITH COPIOUS AMOUNTS OF WATER FOR AT LEAST 15 MINUTES WHILE REMOVING CONTAMINATED CLOTHING AND SHOES.  
 ASSURE ADEQUATE FLUSHING OF THE EYES BY SEPARATING THE EYELIDS WITH FINGERS.  
 IF INHALED, REMOVE TO FRESH AIR. IF NOT BREATHING GIVE ARTIFICIAL RESPIRATION. IF BREATHING IS DIFFICULT, GIVE OXYGEN.  
 IF SWALLOWED, WASH OUT MOUTH WITH WATER PROVIDED PERSON IS CONSCIOUS.  
 CALL A PHYSICIAN.  
 WASH CONTAMINATED CLOTHING BEFORE REUSE.

## - - - - PHYSICAL DATA - - - -

SPECIFIC GRAVITY: 1.135  
 VAPOR DENSITY: 3.4  
 VAPOR PRESSURE: .5 MM @ 20 C  
                   5.5 MM @ 55 C

## APPEARANCE AND ODOR

COLORLESS TO PALE-YELLOW LIQUID

## - - - - FIRE AND EXPLOSION HAZARD DATA - - - -

FLASHPOINT 149 F  
 AUTOIGNITION TEMPERATURE: 915 F  
 LOWER EXPLOSION LEVEL: 1.8%  
 UPPER EXPLOSION LEVEL: 16.3%

## EXTINGUISHING MEDIA

WATER SPRAY.  
 CARBON DIOXIDE, DRY CHEMICAL POWDER OR APPROPRIATE FOAM.

## SPECIAL FIREFIGHTING PROCEDURES

WEAR SELF-CONTAINED BREATHING APPARATUS AND PROTECTIVE CLOTHING TO PREVENT CONTACT WITH SKIN AND EYES.  
 COMBUSTIBLE.

## UNUSUAL FIRE AND EXPLOSIONS HAZARDS

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# M A T E R I A L   S A F E T Y   D A T A   S H E E T

PAGE 4

CUST#: 130451  
 PO#: 54462

PRODUCT #:

NAME: FURFURYL ALCOHOL, 98%

MF: C5H6O2

## - - - - FIRE AND EXPLOSION HAZARD DATA - - - -

FORMS EXPLOSIVE MIXTURES IN AIR.

## - - - - REACTIVITY DATA - - - -

### INCOMPATIBILITIES

ACID CHLORIDES

OXYGEN

AVOID CONTACT WITH ACID.

### HAZARDOUS COMBUSTION OR DECOMPOSITION PRODUCTS

TOXIC FUMES OF:

CARBON MONOXIDE, CARBON DIOXIDE

## - - - - SPILL OR LEAK PROCEDURES - - - -

### STEPS TO BE TAKEN IF MATERIAL IS RELEASED OR SPILLED

EVACUATE AREA.

WEAR SELF-CONTAINED BREATHING APPARATUS, RUBBER BOOTS AND HEAVY

RUBBER GLOVES.

ABSORB ON SAND OR VERMICULITE AND PLACE IN CLOSED CONTAINERS FOR

DISPOSAL.

VENTILATE AREA AND WASH SPILL SITE AFTER MATERIAL PICKUP IS COMPLETE.

### WASTE DISPOSAL METHOD

THIS COMBUSTIBLE MATERIAL MAY BE BURNED IN A CHEMICAL INCINERATOR

EQUIPPED WITH AN AFTERBURNER AND SCRUBBER.

OBSERVE ALL FEDERAL, STATE AND LOCAL ENVIRONMENTAL REGULATIONS.

## - - - - PRECAUTIONS TO BE TAKEN IN HANDLING AND STORAGE - - - -

WEAR APPROPRIATE NIOSH/MSHA-APPROVED RESPIRATOR, CHEMICAL-RESISTANT

GLOVES, SAFETY GOGGLES, OTHER PROTECTIVE CLOTHING.

USE ONLY IN A CHEMICAL FUME HOOD.

SAFETY SHOWER AND EYE BATH.

DO NOT BREATHE VAPOR.

DO NOT GET IN EYES, ON SKIN, ON CLOTHING.

READILY ABSORBED THROUGH SKIN.

WASH THOROUGHLY AFTER HANDLING.

TOXIC.

IRRITANT.

HARMFUL VAPOR.

KEEP TIGHTLY CLOSED.

CONTINUED ON NEXT PAGE

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## M A T E R I A L   S A F E T Y   D A T A   S H E E T

PAGE 5

 CUST#: 130451  
 PO#: 54462

PRODUCT #:

NAME: FURFURYL ALCOHOL, 98%

MF: C5H6O2

- - - - PRECAUTIONS TO BE TAKEN IN HANDLING AND STORAGE - - - -

KEEP AWAY FROM HEAT AND OPEN FLAME.

AIR SENSITIVE

STORE UNDER NITROGEN.

STORE IN A COOL DRY PLACE.

LABEL PRECAUTIONARY STATEMENTS

TOXIC

TOXIC BY INHALATION, IN CONTACT WITH SKIN AND IF SWALLOWED.

IRRITATING TO EYES, RESPIRATORY SYSTEM AND SKIN.

COMBUSTIBLE.

KEEP AWAY FROM HEAT AND OPEN FLAME.

READILY ABSORBED THROUGH SKIN.

SENSITIVE TO AIR

STORE UNDER NITROGEN.

IN CASE OF CONTACT WITH EYES, RINSE IMMEDIATELY WITH PLENTY OF

WATER AND SEEK MEDICAL ADVICE.

IF YOU FEEL UNWELL, SEEK MEDICAL ADVICE (SHOW THE LABEL WHERE POSSIBLE).

DO NOT BREATHE VAPOR/FUMES.

IN CASE OF INSUFFICIENT VENTILATION, WEAR SUITABLE

RESPIRATORY EQUIPMENT.

WEAR SUITABLE PROTECTIVE CLOTHING, GLOVES AND EYE/FACE PROTECTION.

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DOCUMENT 4922

DATE: 11/12/91  
 CUST#: 130451  
 PO#: 15288

# M A T E R I A L   S A F E T Y   D A T A   S H E E T   P A G E   1

## IDENTIFICATION

PRODUCT #: 30519-7  
 CAS #: 100-51-6  
 MF: C7H8O

NAME: BENZYL ALCOHOL, ANHYDROUS, 99+%

## SYNONYMS

BENZAL ALCOHOL \* BENZENECARBINOL \* BENZENEMETHANOL \* BENZOYL ALCOHOL \*  
 HYDROXYTOLUENE \* ALPHA-HYDROXYTOLUENE \* METHANOL, PHENYL- \* NCI-  
 C06111 \* PHENOLCARBINOL \* PHENYL CARBINOL \* PHENYL METHANOL \*  
 PHENYLMETHYL ALCOHOL \* ALPHA-TOLUENOL \*

## TOXICITY HAZARDS

RTECS NO: DN3150000

BENZYL ALCOHOL

## IRRITATION DATA

SKN-MAN 16 MG/48H MLD  
 SKN-RBT 10 MG/24H OPEN MLD  
 SKN-RBT 100 MG/24H MOD  
 EYE-RBT 750 UG OPEN SEV  
 SKN-PIG 100% MOD

CTOIDG 94(8),41,79  
 AMIH8C 4,119,51  
 CTOIDG 94(8),41,79  
 AMIH8C 4,119,51  
 FCTXAV 11,1011,73

## TOXICITY DATA

ORL-RAT LD50:1230 MG/KG  
 IPR-RAT LD50:400 MG/KG  
 IVN-RAT LD50:53 MG/KG  
 IAT-RAT LD50:441 MG/KG  
 ORL-MUS LD50:1580 MG/KG  
 IPR-MUS LD50:650 MG/KG  
 IVN-MUS LD50:324 MG/KG  
 ORL-RBT LD50:1040 MG/KG  
 SKN-RBT LD50:2 GM/KG  
 ORL-BWD LD50:100 MG/KG

FCTXAV 2,327,64  
 NPIRI\* 1,6,74  
 TXAPA9 18,60,71  
 TXAPA9 18,60,71  
 FCTXAV 2,327,64  
 JPMSAE 75,702,86  
 AIPSTAK 135,330,62  
 JPETAB 84,358,45  
 NPIRI\* 1,6,74  
 TXAPA9 21,315,72

## REVIEWS, STANDARDS, AND REGULATIONS

EPA FIFRA 1988 PESTICIDE SUBJECT TO REGISTRATION OR RE-REGISTRATION  
 FEREAC 54,4388,89

NOHS 1974: HZD 11360; NIS 85; TNF 7284; NOS 68; TNE 138757

NOES 1983: HZD 11360000; TNF 128; NIS 19005; NOS 127; TNE 404916; TFE 236470

EPA GENETOX PROGRAM 1988, NEGATIVE: E COLI POLA WITHOUT S9

EPA TSCA CHEMICAL INVENTORY, JUNE 1990

EPA TSCA TEST SUBMISSION (TSCATS) DATA BASE, MARCH 1991

NTP CARCINOGENESIS STUDIES (GAVAGE); NO EVIDENCE: RAT, MOUSE NTPTR\* NTP-TR-343,89

## TARGET ORGAN DATA

BEHAVIORAL (ALTERED SLEEP TIME)  
 BEHAVIORAL (SOMNOLENCE)  
 BEHAVIORAL (TREMOR)

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# M A T E R I A L   S A F E T Y   D A T A   S H E E T      P A G E   2

CUST#: 130451  
 PON#: 15288

PRODUCT #: 30519-7  
 CAS #: 100-51-6  
 MF: C7H8O

NAME: BENZYL ALCOHOL, ANHYDROUS, 99+%

## ----- TOXICITY HAZARDS -----

BEHAVIORAL (EXCITEMENT)  
 BEHAVIORAL (ATAXIA)  
 BEHAVIORAL (COMA)  
 LUNGS, THORAX OR RESPIRATION (CHRONIC PULMONARY EDEMA OR CONGESTION)  
 LUNGS, THORAX OR RESPIRATION (DYSPPNAE)  
 LUNGS, THORAX OR RESPIRATION (OTHER CHANGES)  
 GASTROINTESTINAL (HYPERMOTILITY, DIARRHEA)

ONLY SELECTED REGISTRY OF TOXIC EFFECTS OF CHEMICAL SUBSTANCES (RTECS)  
 DATA IS PRESENTED HERE. SEE ACTUAL ENTRY IN RTECS FOR COMPLETE INFORMATION.

## ----- HEALTH HAZARD DATA -----

### ACUTE EFFECTS

HARMFUL IF SWALLOWED.  
 MAY BE HARMFUL IF INHALED.  
 MAY BE HARMFUL IF ABSORBED THROUGH THE SKIN.  
 VAPOR OR MIST IS IRRITATING TO THE EYES, MUCCOUS MEMBRANES AND UPPER  
 RESPIRATORY TRACT.  
 CAUSES SKIN IRRITATION.  
 CAN CAUSE CNS DEPRESSION.  
 TARGET ORGAN(S):  
 CENTRAL NERVOUS SYSTEM

### FIRST AID

IN CASE OF CONTACT, IMMEDIATELY FLUSH EYES WITH COPIOUS AMOUNTS OF  
 WATER FOR AT LEAST 15 MINUTES.  
 IN CASE OF CONTACT, IMMEDIATELY WASH SKIN WITH SOAP AND COPIOUS  
 AMOUNTS OF WATER.  
 IF INHALED, REMOVE TO FRESH AIR. IF NOT BREATHING GIVE ARTIFICIAL  
 RESPIRATION. IF BREATHING IS DIFFICULT, GIVE OXYGEN.  
 IF SWALLOWED, WASH OUT MOUTH WITH WATER PROVIDED PERSON IS CONSCIOUS.  
 CALL A PHYSICIAN.  
 WASH CONTAMINATED CLOTHING BEFORE REUSE.

## ----- PHYSICAL DATA -----

BOILING PT: 205 C  
 MELTING PT: -15 C  
 SPECIFIC GRAVITY: 1.045  
 VAPOR DENSITY: 3.7  
 VAPOR PRESSURE: 3.75 MM @ 77 C

CONTINUED ON NEXT PAGE



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# M A T E R I A L   S A F E T Y   D A T A   S H E E T PAGE 3

CUST#: 130451  
 PD#: 15288

PRODUCT #: 30519-7  
 CAS #: 100-51-6  
 MF: C7H8O

NAME: BENZYL ALCOHOL, ANHYDROUS, 99+%

## ----- PHYSICAL DATA -----

13.3 MM @ 100 C

APPEARANCE AND ODOR  
 COLORLESS LIQUID

## ----- FIRE AND EXPLOSION HAZARD DATA -----

FLASHPOINT: 213 F  
 AUTOIGNITION TEMPERATURE: 816 F  
 EXTINGUISHING MEDIA  
 WATER SPRAY.  
 CARBON DIOXIDE, DRY CHEMICAL POWDER OR APPROPRIATE FOAM.  
 SPECIAL FIREFIGHTING PROCEDURES  
 WEAR SELF-CONTAINED BREATHING APPARATUS AND PROTECTIVE CLOTHING TO  
 PREVENT CONTACT WITH SKIN AND EYES.  
 UNUSUAL FIRE AND EXPLOSION HAZARDS  
 EMITS TOXIC FUMES UNDER FIRE CONDITIONS.

## ----- REACTIVITY DATA -----

### INCOMPATIBILITIES

STRONG OXIDIZING AGENTS  
 A MIXTURE OF BENZYL ALCOHOL AND 58% SULFURIC ACID DECOMPOSED VIOLENTLY  
 WHEN HEATED TO 180 C. BENZYL ALCOHOL CONTAINING 1.4% HYDROGEN BROMIDE  
 AND 1.1% OF AN IRON(2) SALT POLYMERIZED EXOTHERMALLY WHEN HEATED ABOVE  
 100 C.  
 HAZARDOUS COMBUSTION OR DECOMPOSITION PRODUCTS  
 TOXIC FUMES OF:  
 CARBON MONOXIDE, CARBON DIOXIDE

## ----- SPILL OR LEAK PROCEDURES -----

STEPS TO BE TAKEN IF MATERIAL IS RELEASED OR SPILLED  
 WEAR SELF-CONTAINED BREATHING APPARATUS, RUBBER BOOTS AND HEAVY  
 RUBBER GLOVES.  
 COVER WITH DRY LIME OR SODA ASH, PICK UP, KEEP IN A CLOSED CONTAINER  
 AND HOLD FOR WASTE DISPOSAL.  
 VENTILATE AREA AND WASH SPILL SITE AFTER MATERIAL PICKUP IS COMPLETE.  
 WASTE DISPOSAL METHOD  
 DISSOLVE OR MIX THE MATERIAL WITH A COMBUSTIBLE SOLVENT AND BURN IN A  
 CHEMICAL INCINERATOR EQUIPPED WITH AN AFTERBURNER AND SCRUBBER.

## --- PRECAUTIONS TO BE TAKEN IN HANDLING AND STORAGE ---

CHEMICAL SAFETY GOGGLES.

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# M A T E R I A L   S A F E T Y   D A T A   S H E E T      P A G E   4

CUST#: 130451

PO#: 15288

PRODUCT #: 30519-7

CAS #: 100-51-6

MF: C7H8O

NAME: BENZYL ALCOHOL, ANHYDROUS, 99+%

## --- PRECAUTIONS TO BE TAKEN IN HANDLING AND STORAGE ---

RUBBER GLOVES.

NIOSH/MSHA-APPROVED RESPIRATOR.

SAFETY SHOWER AND EYE BATH.

MECHANICAL EXHAUST REQUIRED.

DO NOT BREATHE VAPOR.

AVOID CONTACT WITH EYES, SKIN AND CLOTHING.

WASH THOROUGHLY AFTER HANDLING.

IRRITANT.

HARMFUL LIQUID.

KEEP TIGHTLY CLOSED.

HYGROSCOPIC

STORE UNDER NITROGEN.

STORE IN A COOL DRY PLACE.

### LABEL PRECAUTIONARY STATEMENTS

HARMFUL

HARMFUL BY INHALATION, IN CONTACT WITH SKIN AND IF SWALLOWED.

IRRITATING TO EYES, RESPIRATORY SYSTEM AND SKIN.

TARGET ORGAN(S):

NERVES

HYGROSCOPIC

HANDLE AND STORE UNDER NITROGEN.

IF YOU FEEL UNWELL, SEEK MEDICAL ADVICE (SHOW THE LABEL WHERE POSSIBLE).

IN CASE OF CONTACT WITH EYES, RINSE IMMEDIATELY WITH PLENTY OF WATER AND SEEK MEDICAL ADVICE.

TAKE OFF IMMEDIATELY ALL CONTAMINATED CLOTHING.

WEAR SUITABLE PROTECTIVE CLOTHING, GLOVES AND EYE/FACE PROTECTION.

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# American Burdick & Jackson

## Material Safety Data Sheet

emergency telephone no. 312/973-3600 (American Scientific Products)

chemtrec telephone no. 800/424-9300

information telephone no. 616/726-3171 (American Burdick &amp; Jackson)

### MATERIAL SAFETY DATA SHEET

#### I. Identification

chemical name N-Methylpyrrolidone molecular weight 99.13

chemical family Cyclic Amide formula C<sub>5</sub>H<sub>9</sub>NO

synonyms NMP, N-Methyl-2-Pyrrolidone

DOT proper shipping name Combustible Liquid, n.o.s.

DOT hazard class Combustible Liquid

DOT identification no. NA1993 CAS no. 872-50-4

N-METHYLPYRROLIDONE

#### II. Physical and Chemical Data

boiling point, 760mm Hg. 202°C freezing point -24.4°C evaporation rate (BuAc = 1) ca 0.1

vapor pressure at 20°C &lt; 1 mm Hg vapor density (air = 1) 3.4 solubility in water Complete

% volatiles by volume ca 100 specific gravity (H<sub>2</sub>O = 1) @ 25°C 1.028 stability Stable

hazardous polymerization Not expected to occur.

appearance and odor Clear, colorless liquid with a slight amine odor.

conditions to avoid Heat, sparks, open flame, open containers, and poor ventilation.

materials to avoid Strong oxidizing agents and strong acids.

hazardous decomposition products Incomplete combustion can generate carbon monoxide, nitrogen oxides, ammonia, and other toxic vapors.

#### III. Fire and Explosion Hazard Data

flash point, (test method) 93°C (PM closed cup) auto ignition temperature 346°C

flammable limits in air % by volume: lower limit 0.9 upper limit 3.9

unusual fire and explosion hazards None, other than combustibility.

extinguishing media Carbon dioxide, dry chemical, or foam.

special fire fighting procedures Wear full protective clothing and self-contained breathing apparatus. Heat will build pressure and may rupture closed storage containers. Keep fire-exposed containers cool with water spray.

#### IV. Hazardous Components

N-Methylpyrrolidone % ca 100 TLV not listed CAS no. 872-50-4

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Hospital Supply Corporation1953 South Harvey Street  
Muskegon MI 49442

**V. Health Hazards**Occupational Exposure Limits

OSHA	8-hour PEL	-	not listed
	Ceiling	-	not listed
	Peak	-	not listed

Concentration Immediately Dangerous to Health

OSHA/NIOSH	not listed
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Odor Threshold

ACGIH	TLV-TWA	-	not listed
	TLV-STEL (15-min)	-	not listed

NSC & OHS	not listed
NIOSH	not listed

NIOSH	TLV-TWA	-	not listed
	TLV-C	-	not listed

Primary Routes of Entry

N-Methylpyrrolidone may exert its effects through inhalation, skin absorption, and ingestion.

Industrial Exposure: Route of Exposure/Signs and Symptoms

Inhalation: Exposure can cause slight irritation to mucous membranes.

Eye Contact: Liquid and high vapor concentration can be irritating. Direct contact may cause corneal damage.

Skin Contact: Prolonged or repeated skin contact can cause irritation.

Ingestion: Can cause gastrointestinal tract discomfort, diarrhea, and difficult breathing.

Effects of Overexposure

N-Methylpyrrolidone is an eye and slight mucous membrane irritant. Systemic effects information is inadequate/incomplete.

Medical Condition Aggravated by Exposure

Preclude from exposure those individuals susceptible to dermatitis.

Emergency First Aid

- Inhalation: Immediately remove to fresh air. If not breathing, administer mouth-to-mouth rescue breathing. If there is no pulse administer cardiopulmonary resuscitation (CPR). Contact physician immediately.
- Eye Contact: Rinse with copious amounts of water for at least 15 minutes. Get emergency medical assistance.
- Skin Contact: Flush thoroughly for at least 15 minutes. Wash affected skin with soap and water. Remove contaminated clothing and shoes. Wash clothing before re-use, and discard contaminated shoes. Get emergency medical assistance.
- Ingestion: Call local Poison Control Center for assistance. Contact physician immediately. Never induce vomiting or give anything by mouth to a victim unconscious or having convulsions.

**VI. Safety Measures and Equipment**

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- Ventilation: Adequate ventilation is required to protect personnel from exposure to chemical vapors and to minimize fire hazards. The choice of ventilation equipment, either local or general, will depend on the conditions of use, quantity of material, and other operating parameters.
- Respiratory: Use approved respirator equipment. Follow NIOSH and equipment manufacturer's recommendations to determine appropriate equipment (air-purifying, air-supplied, or self-contained breathing apparatus).
- Eyes: Safety glasses are considered minimum protection. Goggles or face shield may be necessary depending on quantity of material and conditions of use.
- Skin: Protective gloves and clothing are recommended. The choice of material must be based on chemical resistance and other user requirements. Generally, neoprene offers acceptable chemical resistance. Individuals who are acutely and specifically sensitive to N-methylpyrrolidone may require additional protective equipment.

Storage: N-Methylpyrrolidone should be protected from temperature extremes and direct sunlight. Proper storage of N-methylpyrrolidone must be determined based on other materials stored and their hazards and potential chemical incompatibility. In general, N-methylpyrrolidone should be stored in an acceptably protected and secure general storage room.

Other: Emergency eye wash fountains and safety showers should be available in the vicinity of any potential exposure

## VII. Spill and Disposal Data

Spill Control: Protect from ignition. Wear protective clothing and use approved respirator equipment. Absorb spilled material in an absorbent recommended for solvent spills and remove to a safe location for disposal by approved methods. If released to the environment, comply with all regulatory notification requirements.

Waste Disposal: Although N-methylpyrrolidone is not classified as an EPA hazardous waste, it should be disposed of in an environmentally acceptable manner.

Revision Date: 10/85

### KEY

ca	Approximately	STEL	Short Term Exposure Level
na	Not applicable	TLV	Threshold Limit Value
C	Ceiling	TWA	Time Weighted Average
PEL	Permissible Exposure Level	BuAc	Butyl Acetate
NSC	National Safety Council ("Fundamentals of Industrial Hygiene", 1983)		
OHS	Occupational Health Services ("Hazardline")		

# REPORT

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Identification of Biodegradable,  
Environmentally Compatible

Methods for Epoxy Removal

— Phase II

To

Aerospace Guidance and Metrology

Center (AGMC)

Newark Air Force Base, Ohio

February, 1995

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## Executive Summary

The Aerospace Guidance and Metrology Center (AGMC) located at Newark Air Force Base (NAFB) repairs and maintains navigation and guidance equipment for the United States Air Force and other components of the Department of Defense. Epoxy resin based adhesives are used to bond components of this equipment. During repair, the adhesives are removed and the surfaces are cleaned before they are repaired and rebonded. Current methods used for removing epoxy adhesive involve heat, mechanical scrapping, stripping by oxidation and degradation and bead blasting. The chemicals used for adhesive stripping, such as nitric acid and methylene chloride, are corrosive and environmentally undesirable. The mechanical methods have the potential for damaging the parts.

Battelle carried out a two-phase research program for AGMC to develop biodegradable, environmentally compatible methods for epoxy removal. In Phase 1, Battelle used results from the literature and contacts with suppliers of commercial methods and solvents to define a preliminary set of solvent characteristics for epoxy removal. This was followed by identification of candidate solvents using a solubility parameter methodology. The results of swelling tests with 14 candidate solvents showed that N-methyl pyrrolidone (NMP), benzyl alcohol (BA), and furfuryl alcohol (FA) provided the best potential for removing epoxy resins. It was found that the addition of water (10 to 15 percent) to the solvent candidate promoted the epoxy resin removal process.

The objective of Phase II was to develop an epoxy removal process based on the best of the solvent-water combinations identified in Phase 1. A number of candidate additives including surfactants, epoxy degradation agents and corrosion inhibitors were also identified. Stainless steel coupons coated with LCA4/BA5 epoxy adhesive and cured for 6 hours at 200 F were used for testing. Statistically designed grids of experiments were carried out to identify the most effective additives. N-methyl pyrrolidone with nonionic surfactants, citric acid catalyst, and the sodium salt of anthraquinone sulfonic acid provided the best formulation. Mechanical agitation and sonication did not show significant improvement in cleaning efficiency. It was also found that cleaning should be carried out at 75 C or above to complete the cleaning operation in less than 8 hours.

The candidate cleaning solutions were tested for metal compatibility using six metallic substrates including beryllium. All metals but chromium steel were compatible with all solutions as demonstrated by the absence of significant weight change, pitting, or gas evolution on 16 hour soaking times. Chromium steel may be compatible with our preferred cleaning solution using shorter ( $< 8$  hours) soaking times but this needs confirmation.

Battelle used the experimental results to develop a prototype epoxy removal process which included equipment recommendations, cleaning solution preparation specifications, cleaning procedures, quality assurance, and disposal methods for used solvents. A liter of the NMP-water cleaning solution was also forwarded to AGMC for cleaning trials.

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## **1.0 Introduction**

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### **1.1 Background**

The Aerospace Guidance and Metrology Center (AGMC), located at Newark Air Force Base (NAFB), Ohio, repairs and maintains navigation and guidance equipment for the United States Air Force and other components of the Department of Defense. Thousands of these sophisticated electromechanical devices are repaired each year at the Center.

Epoxy resins are widely used in the assembly of these parts as adhesives because of their high temperature resistance, excellent strength and good resistance to chemicals. During the repair of parts and components it is frequently necessary to remove the adhesive. Since the thermoset epoxy adhesive is crosslinked, it is difficult to remove. Current methods of removing epoxy resins involve the use of heat, mechanical scraping, stripping by oxidation and degradation, and bead blasting. Many of these operations, such as mechanical scraping or bead blasting, can cause surface damage to the parts. Current operations also use chemical stripping based on chemicals which are toxic and/or environmentally undesirable (e.g., cause smog or deplete the earth's ozone layer).

AGMC is committed to reducing the use of hazardous chemicals and improving its repair processes. Therefore, the objective of this two-phased program was to identify alternative, solvent-based methods for epoxy removal which are efficient, more environmentally compatible than those in current processes; to experimentally evaluate these methods, and to develop a prototype process for epoxy removal. The process must be safe, should not degrade the reliability of the equipment being repaired and maintained by AGMC, and must be environmentally friendly. Standards of comparison include current epoxy removal processes that use dimethyl sulfoxide (DMSO) and nitric acid, methyl ethyl ketone, methylene chloride, chlorofluorocarbons (CFC's), and mechanical scraping.

## **1.2 Summary of the Work Carried Out in Phase I**

The objective of Phase I of this program was to identify potential solvent-based methods for effectively removing epoxy adhesives in an immersion/soaking process. A focused literature search and adhesive supplier contacts were used to define a preliminary set of candidate solvent systems, including commercially available products. A theoretical solubility parameter model was then used to screen the candidates.

Fourteen potential solvents and solvent systems were identified for preliminary feasibility testing. Swelling tests were carried out for one epoxy adhesive and the 14 candidate solvents. It was discovered that the addition of water to several of the solvents created an excellent epoxy stripping media. The three solvents showing the most potential, namely N-methyl pyrrolidone, benzyl alcohol, and furfuryl alcohol were chosen for epoxy removal tests. These tests were carried out for the three neat solvent systems and solvent/water mixtures using aluminum coupons coated with seven different epoxies in common use at AGMC. All the tested epoxies were easily removed by one or more of the water-solvent mixtures after 24 hour exposure at 70 C. Epoxy removal tests with sample parts supplied by AGMC were carried out to confirm the performance of these solvent mixtures with realistic substrate materials and geometries. Parts testing confirmed the overall effectiveness of the three solvents if water was added.

The results from Phase I were sufficiently promising to recommend that a second phase of the program be carried out to finalize the development of an alternate epoxy removal process.

## **1.3 Formulation of a Solvent System**

Epoxy removal may be construed as a three-step process. In Step 1, the solvent or solvent mixture diffuses into and wets the interface between the metal and adhesive. In the second step, the solvent system dissolves the epoxy. The concentrated solution is then removed by thermal mixing with the bulk of the solvent during the final step.

For the epoxy removal process to be efficient and effective, it is important that the solubilizing capacity of the solvent be augmented by additives such as surfactants and wetting

agents, catalysts for depolymerizing the cured epoxy, and corrosion inhibitors. Whereas the first two additives will help the solvent system in maintaining homogeneity of the solution and in wetting the metal interface, the third additive, namely the catalyst, will break up the crosslinked matrix and reduce the molecular weight of the resin making it more amenable to dissolution by the solvent. The corrosion inhibitor is required to inhibit metal corrosion, particularly in the presence of water. For the additives to be helpful and not harmful to the epoxy removal system, they should not only be of proper chemical type but in proper chemical proportion. Hence, it is essential to identify the useful additives for the solvent system and optimize the composition for the most effective removal of epoxy resin.

#### **1.4 Objectives of Phase II Program**

The objective of Phase II was to identify the components and optimize the composition of a solvent immersion stripper system which is environmentally compatible, noncorrosive to the metal substrates, and safe for routine use at AGMC. It was also desired that the process be effective in a time period less than 8 hours to permit treatment in one shift.

#### **1.5 Technical Approach**

To accomplish the objectives of the Phase II program, a detailed work plan was prepared and submitted to AGMC. The work plan included six tasks as follows:

- Formulate environmentally-compatible solvent systems by incorporating additives to solvents selected in Phase I.
- Examine the compatibility of the solvent systems with the various substrate metals used in typical parts.
- Optimize the composition of the candidate solvent system and the physical parameters involved in the cleaning process.
- Confirm cleaning process performance by cleaning actual parts.
- Develop and document a prototype process for delivery to AGMC.



- Provide progress reports and final report including all relevant data obtained in the program.

## **2.0 Task Results**

As mentioned earlier, the solvent system should have additives including a wetting agent, an epoxy depolymerization catalysts and a corrosion inhibitor. The objective of this task was to identify suitable additives for the three candidate solvent systems and to assess their efficiency in cleaning epoxy-coated steel coupons.

### **2.1 Evaluating Cleaning Efficiency**

Four different methods were examined for monitoring cleaning efficiency. They included weight loss, microscopic examination, infrared spectrophotometry and analysis of surface cleanliness using a surface quality monitor. The following summarizes the results obtained by the four methods.

**Residual Weight Loss Method.** The weight loss method was evaluated using steel panels coated with epoxy resin. The panels were cleaned, dried, and weighed to  $\pm 0.0002$  gm. The panels ( $\approx 10$  to  $11 \text{ cm}^2$ ) were then coated with epoxy adhesive LCA4/BA5, cured at 200 F for 4 hours and aged at room temperature for 24 hours. The panels were cooled to room temperature and coating weights were measured. This was followed by cleaning the panels using the three cleaning solutions mentioned earlier and immersing the coated panels for 24 hours at 70 C. The cleaned panels were washed and rinsed with distilled water and dried in an air oven at 200 F for 2 hours. Panels were then cooled to room temperature in a desiccator and weighed to  $\pm 0.0002$  gm. The difference between the final and initial weight gave a measure of epoxy residue left on the panel as a measure of cleaning efficiency. The results (Table 1) indicate that the cleaning efficiency of the solutions can be quantitatively measured using this technique.

**Table 1. Cleaning Efficiency by Residual Weight and Microscopic Examination**

Cleaning Solution	Initial Coating Weight, mg	Cleaning Efficiency		
		Average Residual Weight, %	Microscopic Evaluation	Cleaning Efficiency
FA/Water	7.2	0.000	0	Complete removal on wiping
NMP/Water	7.0	0.036	22	ditto
BA/Water	8.4	0.030	1	"

FA = Furfuryl alcohol  
 NMP = N-methyl pyrrolidone  
 BA = Benzyl alcohol.

**Microscopic Examination.** The microscopy work was carried out using both aluminum and steel coupons coated with epoxy adhesives, LCA4/BA5 and FA8/BA5. Approximately 2 to 3 mil thick coatings were laid down on the coupons using a doctor blade. The adhesives were cured at 200 F for 4 hours. The cured coupons were immersed in the cleaning solutions made out of N-methyl pyrrolidone (NMP), benzyl alcohol (BA), or furfuryl alcohol (FA) containing water for a period of 24 hours at 70 C. The cleaned surfaces were inspected using an Olympus (BHZVMA Mode) microscope and photographs were taken at 50x magnification. Initial results indicated that the microscopic examination data agrees qualitatively with the residual weight measurements. To make the results more quantitative, the coated area was divided into 32 squares. Each square was examined using the optical microscope and given a cleanliness rating from 0 to 5, 0 being total removal and 5 being a completely intact coating. The sum of the grading marks was used as a measure of cleaning efficiency.

Table 1 compares the results of microscopic inspection with the results from residual weight measurement. Although the results obtained by the two methods agree in general, there are some discrepancies. Further examination showed that the height (thickness) of the

residual adhesive is not the same in all locations. The residual weight measurement takes into consideration the total amount of material left, thick or thin. The microscopic examination on the other hand looks for the clean surface and does not take into account the thickness of the adhesive left on the surface. Since complete removal of epoxy adhesives is the main focus of this program, the microscopic method was used to complement the residual weight measurement to evaluate the cleaning efficiency of solvent formulations.

**Infrared (IR) Method.** Grazing angle reflectance was used to examine the sample surfaces. This is an infrared technique that focuses the infrared beam onto the surface of the sample at a variable angle of incidence. The reflected beam is then collected and results in an infrared spectrum of the materials on the surface of the sample. In these preliminary experiments, the angle of incidence was  $80^\circ$ . The spectra shown in Figures 1 and 2 were obtained from samples cleaned by two different solutions, namely NMP/water and BA/water. The infrared absorption bands (represented as % transmittance) in Figure 1 are greater in intensity than those in Figure 2, possibly indicating that more residue is left after cleaning the first solution than after cleaning with the second one. The bands in these spectra are characteristic of the epoxy used in the coating process. The band occurring near  $1650\text{ cm}^{-1}$  suggests that the material was cured with an amine curing agent. Other bands near  $1020\text{ cm}^{-1}$ ,  $1240\text{ cm}^{-1}$ ,  $1600\text{ cm}^{-1}$  and  $1500\text{ cm}^{-1}$  represent the epoxy resin. Quantitative measurement of the area under the peaks could be used to quantify the uncleaned area. The use of varying angles of incidence and polarized light are typically used to optimize this technique for a particular system. However, this IR technique could not be used as a quantitative method because the spectra only reflects the epoxy surface and does not take into account the varying thickness of the residual epoxy layer. In addition, the interference of solvent bands with epoxy bands may cause problems.

**Surface Quality Monitor (SQM) Technique.** This technique measures the number of photoelectrons emitted from the cleaned portion of the metal surface vis-a-vis the uncleaned surface that does not emit any photoelectrons. This technique is, therefore, a very sensitive method for measuring cleaning efficiency. However, like IR, this could not be

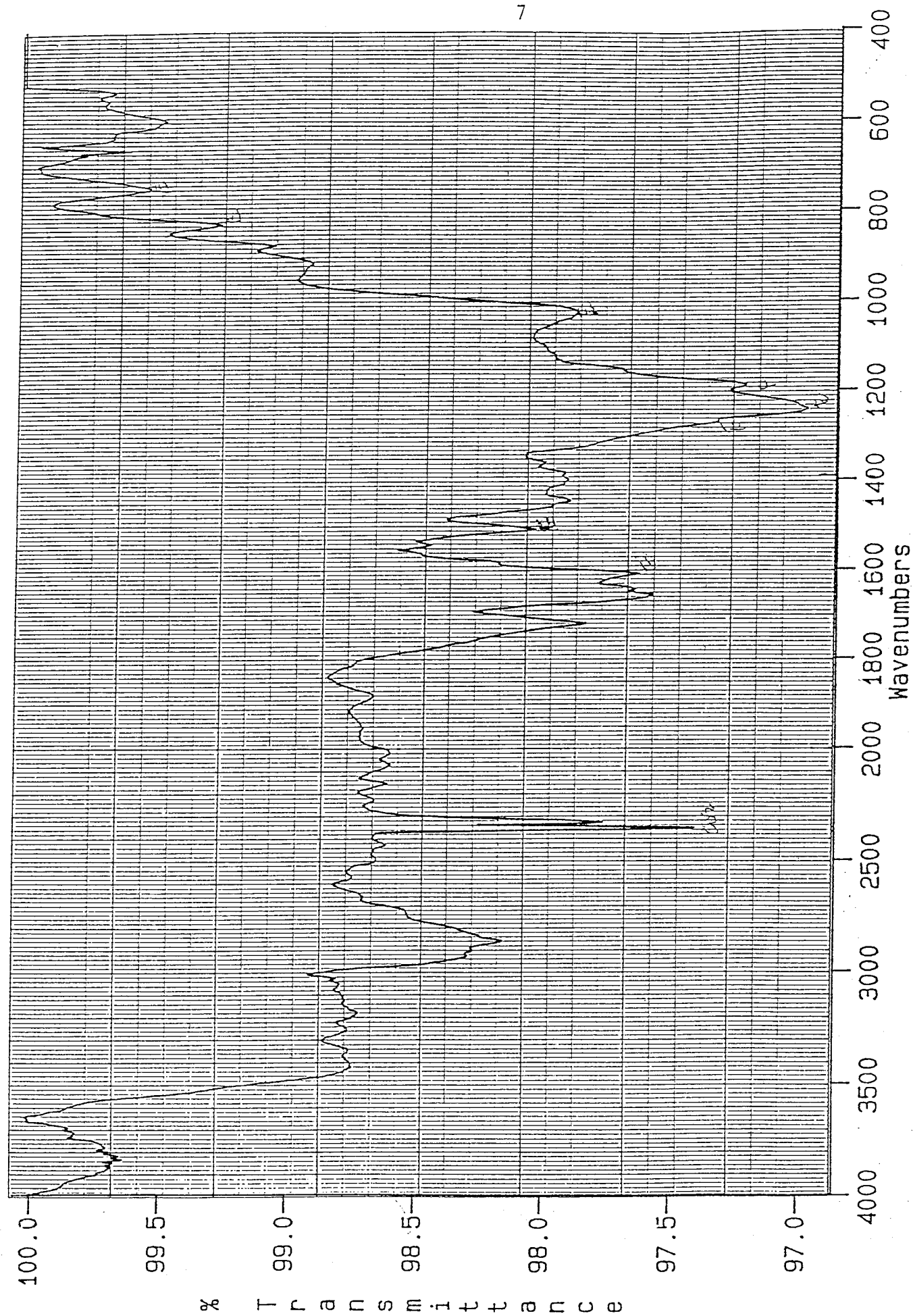


Figure 1. Infrared Spectra of an Epoxy Coated Steel Panel Cleaned With FA/Water Solution

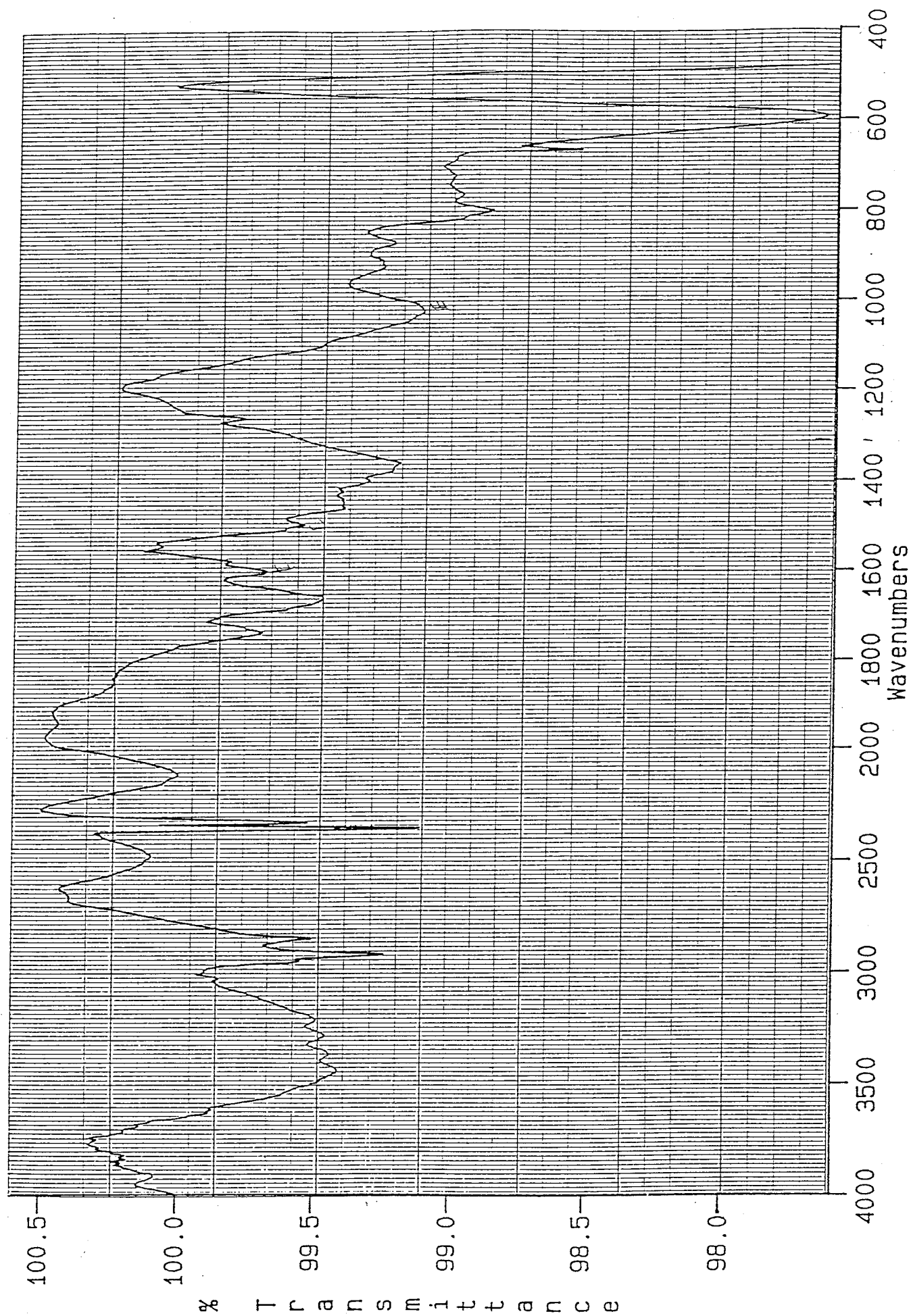


Figure 2. Infrared Spectra of an Epoxy Coated Specimen After Cleaned With BA/Water Solution

made into a quantitative technique since the intensity of photoelectrons captured by the detector depends not only on the extent of the clean surface but also on the distance of the receiver from the surface. It was very difficult to maintain constant distance due to the varying thickness of the residual epoxy.

Based on the results obtained in this task, it was decided to use the combination of microscopic technique and residual weight measurement for measuring cleaning efficiency of the formulated solvent systems.

## **2.2 Selection of Additives**

As mentioned earlier, the cleaning solution may use additives to boost the cleaning efficiency of the candidate solvents. A limited search was carried out to identify solvent formulations of paint and adhesive stripping agents. Selected solvent manufacturers were also contacted to provide information on the formulation of the stripping agents. The following summarizes the results of the information survey and the selection of candidate additives.

In a 1986 patent<sup>(1)</sup> assigned to the U.S. Navy, Peter J. Hearst describes an epoxy stripping solution in which he used Igepal C0630 (polyethylene glycol ether of nonyl phenol) and Tergitol NP-9 (a nonylphenol ethoxylate) as surfactants along with a mixture of N-methyl pyrrolidone and ethanolamine as solvent.

In a Japanese patent<sup>(2)</sup> assigned to Fuji Chemical, Nishibu, there is a description of a process to remove epoxy resins from transfer printing plates using solvents such as cyclohexanone, trichloroethane, along with additives such as phosphoric acid and propylene-ethylene-glycol nonylphenyl ether. Cresylic acid and formic acid have been used as epoxy degradation catalysts.

Beck Chemical<sup>(3)</sup> uses a mixture of methylene chloride and formic acid with phenol and surfactants in their epoxy stripper T251-C.

The epoxy stripper of Miller Stevenson Co.<sup>(4)</sup> MS-111 contains methylene chloride, phenols and organic acids.

Dow Chemical uses NMP and glacial acetic acid in their epoxy stripping solvent system<sup>(5)</sup>.

Bacon Industries "Epoxy Strip T251-C" contains methylene chloride and glacial acetic acid as main components<sup>(6)</sup>.

"Industrial Surfactants" authored by Earnest W. Flick (Noyes Publications, Park Ridge, New Jersey, USA, 1988) describes commercially available surfactants used for a variety of applications from textile sizing to metal cleaning. The surfactants used for metal cleaning were selected since they are likely to wet the metal surface better than the ones used to clean other surfaces.

The Chemical Encyclopedia<sup>(7)</sup> lists corrosion inhibitors which can be added in small quantity to the cleaning solution to prevent corrosion of metals.

The information obtained from this search was used to identify suitable additives for cleaning solutions. Care was taken to select additives that were inert with respect to the candidate solvent water mixtures. The selected additives are listed in Tables 2 and 3.

In the past, acid catalysts have been used for degrading and dissolving epoxy resins and in formulating stripping agents. Three acids, lactic acid, citric acid and p-toluene sulfonic acid, were selected as epoxy degradation catalysts for this program. It was anticipated that this would effectively hydrolyze ester, urethane and amino alcohol crosslinks as well as disrupt bonds with metal oxide layers on the metallic substrates.

### **2.3 Preparation of Epoxy Coated Panels**

**Surface Treatment.** Two alternative methods of surface treatment of steel panels were recommended, one based on dilute nitric acid by Bacon Industries and the other based on sodium dichromate-sulfuric acid by the Military Standardization Handbook, MIL HDBK-691A, 17 May 1965. Both methods were tried. The coupons treated with chromic acid appeared cleaner and brighter than the coupons treated with nitric acid. Hence, chromic acid was used in the surface preparation of all the steel coupons.

A hole was drilled in each coupon, followed by cleaning with acetone. The coupons were then immersed in a solution containing 15 g sodium dichromate, 80 cc concentrated

**Table 2. Selected Surfactants for Epoxy Removal Study**

Trade Name	Chemical Type	Supplier	Relevant Application
<b>Surfactant</b>			
Igepal CO 520, and CO630	Nonyl phenoxy poly-(ethyleneoxy) ethanol	GAF Corp.	Wetting, detergency
Tetronic	Block copolymers of ethylene oxide/propylene oxide and ethylene diamine	BASF Corp.	Metal cleaning
Antarox BL-240	Modified linear aliphatic polyether (biodegradable)	GAF Chemical Corp.	Metal spray cleaning, aircraft cleaning, rapid wetting
Tergitol NP-9	Nonyl phenol ethoxylate	Union Carbide	Paint stripping
Trylon 3300	Anionic ether	Emery Industries	Versatile emulsifier, used in cleaning metal parts, engine blocks
ATSURF F-12	Phosphoric ester type anionic fluorosurfactant	ICI America Inc.	Strongly adheres to metal surface
Pluorfac B25-5	Linear alcohol ethoxylate	BASF	Wetting agent in high mechanical energy cleaning procedures
Triton 50	Quaternary salt of long-chain fatty acids	Union Carbide	Excellent wetting and dispersing agent
GAFAC RM410	Free acid of phosphate ester	GAF	Metal cleaning

**Table 3. Selected Corrosion Inhibitors for Epoxy Removal Study**

Chemical Type
Methyl ethyl pyridine
Naphthyl amine
Butyl mercaptan
Anthraquinone disulfonic acid (sod. salt)
Dodecyl sulfate (sod. salt)
1-Naphthol 3,6-disulfonic acid



sulfuric acid and 450 cc distilled water for 5 minutes at 70 C, after which they were taken out and rinsed in tap water followed by thorough rinsing with distilled water. The specimens were then dried at 70 C for approximately 10 minutes. Individual coupons were then identified by engraving a number at the top and weighed individually using a Metler balance.

**Adhesive Coating.** The adhesive was prepared by mixing 4.5 parts of BA5 with 100 parts of LCA4. Approximately 2 inch by 1 inch of the coupon surface was coated with adhesive using a stainless steel drawdown rod. A second coat was applied 5 minutes after the first coating to cover the surface thoroughly and avoid any discontinuity on the coating surface. The coupons were then baked at 200 F for 4 hours in an air oven. After they were cooled, they were weighed again and the coating thickness was measured using a microgauge (Minitest 1000S, Electro-physik, Koln, Germany). A second coat was applied and cured for an additional 4 hours. At the end, the thickness increased from 5 mil to approximately 10 mils. The glass transition temperature of the epoxy adhesive was measured using a Differential Scanning Calorimetry (DSC) to evaluate the state of cure. DSC of the specimens cured for 4 hours showed an endotherm and initial Tg at 48 C indicating that the epoxy was not fully cured. Hence, the samples were cured for 2 more hours at 200 F. The specimens did not show any endotherm and the major Tg was approximately 104 C to 105 C. Hence, all specimens were routinely cured for 6 hours at 200 F.

## **2.4 Cleaning System Formulation of Screening Experiments**

**Screening Experiments.** Since the amount of water is an important variable in the statistical design of experiments, it was important to find out the maximum and minimum percentage of water that could be used for epoxy removal. Both 0 percent and 30 percent water were tried following the standard procedure of epoxy removal (70 C immersion for 24 hours). None of the three solvents (Benzyl alcohol, N-Methyl pyrrolidone and Furfuryl alcohol) alone did a good job on epoxy removal. With 30 percent water, the coating lifted up completely when immersed in benzyl and furfuryl alcohol but not when N-methyl

pyrrolidone was used as epoxy removal solvent. Thirty percent water was probably too high for NMP.

**Surfactants and Corrosion Inhibitors.** The screening of candidate surfactants and corrosion inhibitors was carried out using a statistically designed grid of experiments. Table 5 lists the 12 trials carried out to look at the different types of solvents, surfactants, and corrosion inhibitors. In this case, the same quantity of surfactant and corrosion inhibitor were used for each trial. Also, the quantity of water (14.5 weight percent) was kept the same for each trial. The corrosion inhibitors were designated as A, B, and C and the three surfactants were designated as anionic, nonionic and cationic in the experimental grid. They are described in Table 4.

**Table 4. Surfactants and Corrosion Inhibitors Used in First Screening Experiments**

Surfactant		Inhibitor	
Nonionic	Antarox BLI-240	Basic (A)	5-Ethyl-2-methyl pyridine
Cationic	Triton RW-50	Acidic (C)	Anthraquinone-2-sulfonic acid, sodium salt
Anionic	Gafac RM-410	Salt (B)	Dodecyl sulfate, sodium salt

The individual cleaning solutions contained 84 percent solvent, 14.5 percent distilled water, 1.0 percent surfactant, and 0.5 percent corrosion inhibitor. The three solvents used in the cleaning experiments were furfuryl alcohol, n-methyl pyrrolidone, and ethyl lactate. Ethyl lactate is a biodegradable solvent that is compatible with the environment. The mixture of ethyl lactate and water was found to be as effective as benzyl alcohol for the epoxy removal process. For that reason, ethyl lactate was substituted for benzyl alcohol in the Phase II study.

Triplicates of epoxy coated stainless steel coupons were exposed to each of the solutions mentioned above for 24 hours at 70 C. The epoxy adhesive used in this experiment consisted of LCA4/BA5 cured for 6 hours at 200 F. The weights of the bare metal coupons and that of the coated coupons as well as their thicknesses were measured.

In most cases, the coating lifted off. Parts of the coating left behind was removed by wiping the specimen with Kimwipe. The panels were then washed with distilled water followed by acetone and dried in an air oven at 70 C.

The weights of individual dry panels were then measured. Table 5 provides the average residual weight after cleaning with a particular cleaning solution. The results of microscopic evaluation are also presented in this table. The results indicate that all the solutions (including the controls) except solution #6 (FA/Anionic/B) are very efficient in removing epoxy adhesive, particularly the ones based on NMP. The difference in surfactant type and corrosion inhibitor type did not have a significant influence on the results. Combinations of EL with anionic and nonionic surfactants are somewhat more efficient than that with the cationic surfactant. Also, the mixture of FA with the nonionic surfactant was more effective than FA with the anionic surfactant.

## **2.5 Solvent Systems with Epoxy Degradation Catalyst**

Epoxy degradation catalysts were added to the cleaning solutions and their efficiency was measured in conjunction with surfactants and corrosion inhibitors. Panels coated with epoxy adhesive LCA4/BA5 and cured for 6 hours were cleaned by soaking the coupons in the cleaning system for 24 hours at 70 C followed by exposure to ultrasonic agitation for 5 minutes. The statistically designed grid of 12 cleaning solution formulations along with 3 controls was used for cleaning the panels. Cleaning efficiency was measured both by residual weight and microscopic examination. The composition of the solutions along with the results of cleaning efficiency are given in Table 6. Both sets of data were taken into consideration in the statistical analysis of the results. The analysis showed that whereas p-toluene sulfonic acid is the best catalyst, the nonionic surfactant provides best wettability and ethyl-methyl pyridine or sodium salt of anthraquinone sulfonic acid acts as the best corrosion inhibitor. NMP appears to be superior to ethyl lactate or furfural alcohol as the solvent. The best stripper components based on NMP, for the removal of LCA4/BA5 epoxy is given in Table 7.

**Table 5. Residual Weight and Cleaning Efficiency of Solvent Mixtures on LCA4/BA5 Epoxy**

Solution Number	Cleaning Solution <sup>(a)</sup>	Cleaning Efficiency		
		Average Residual Weight, percent	Microscopic Evaluation <sup>(b)</sup>	Cleaning Efficiency <sup>(c)</sup>
Control	FA/Water	0.000	0	Complete removal on wiping
Control	NMP/Water	0.036	22	ditto
Control	EL/Water	0.030	1	"
1	NMP/Anionic/A	0.000	36	Complete removal by dispersing into cleaning solution
2	EL/Nonionic/A	0.015	8	ditto
3	FA/Cationic/A	0.037	9	"
4	NMP/Nonionic/B	0.075	19	"
5	EL/Cationic/B	0.045	4	Half epoxy intact in one panel, lifted in 2 others
6	FA/Anionic/B	0.389	48	Softened on panel, removed by wipe
7	NMP/Cationic/C	0.025	19	Lifted off and dispersed into solution
8	EL/Anionic/C	0.037	7	ditto
9	FA/Nonionic/C	0.041	6	"
10	FA/Cationic/A	0.060	14	"
11	EL/Anionic/C	0.014	1	"
12	NMP/Anionic/A	0.000	2	"

- (a) FA = furfuryl alcohol, NMP = N-methyl pyrrolidone, EL = ethyl lactate (84 percent solvent, 14.5 percent water, 1 percent surfactant, and 0.5 percent corrosion inhibitor).
- (b) Points assigned to viewing area; point range 0 for no residue to 160 for total coverage.
- (c) After 24 hours submersion in cleaning solution at 70 C.

**Table 6. Residual Weight and Cleaning Efficiency of Solvent Mixtures on LCA4/BA5 Epoxy Cured at 6 Hours at 200 F**

Solution Number	Cleaning Solution <sup>(a)</sup>	Cleaning Efficiency		
		Average Residual Weight, percent <sup>(b)</sup>	Microscopic Evaluation <sup>(c)</sup>	Visual Observation <sup>(d)</sup>
13 Control	NMP/Water	0.064	15	Lifted off and dispersed into solution
14 Control	EL/Water	0.031	2.5	ditto
15 Control	FA/Water	0.085	4.5	"
16	NMP/anionic/A/lactic acid	0.059	5.5	"
17	EL/nonionic/A/PTSA	56.929	160 (total coverage)	Partially removed by wiping
18	FA/cationic/A/citric acid	0.042	5.5	Lifted off and dispersed into solution
19	NMP/nonionic/B/citric acid	0.000	0.5	ditto
20	EL/cationic/B/lactic acid	83.800	160 (total coverage)	Not removed by wiping
21	FA/anionic/B/PTSA			Mixture polymerized
22	NMP/cationic/C/PTSA	0.036	4.5	Soften on panel, removed by wiping
23	EL/anionic/C/citric acid	98.876	160 (total coverage)	Not removed by wiping
24	FA/nonionic/C/lactic acid	0.130	2.5	Lifted off and dispersed into solution
25	FA/cationic/A/citric acid	0.069	5	ditto
26	EL/anionic/C/citric acid	72.763	160 (total coverage)	Partially removed by wiping
27	NMP/anionic/A/lactic acid	0.080	5.5	Lifted off and dispersed into solution
38	EL/nonionic/A	0.000	2	Complete removal by wiping on 2; incomplete on 1
39	NMP/nonionic/A/PTSA	4.966	28	Complete removal on 1; incomplete on 2

(a) NMP = N-Methyl pyrrolidone, EL = ethyl lactate, FA = furfuryl alcohol, PTSA = p-toluene sulfuric acid.

(b) Average of better two (2) values of triplicate results.

(c) Points assigned to viewing area; point range 0 for no residue to 160 for total coverage.

(d) After 24 hrs submersion in cleaning solution at 70 C.

Approximate pH of each cleaning solution was measured using pH papers. The pH of all solutions were lower than seven. pH does not appear to influence the cleaning efficiency to any appreciable extent for the NMP-based system. However, the addition of acid to ethyl lactate-water cleaners appears to decrease its capacity to remove the epoxy coatings.

**Table 7. Effective Cleaning Solutions**

[Solvent/Water/Catalyst/Surfactant/Inhibitor (82:14:2.5/1.0/0.5%)]

Number	Solvent	Co-Solvent	Catalyst	Surfactant	Corrosion Inhibitor
1	N-Methyl Pyrrolidine	Water	Citric Acid	Atarax AL-240	Anthraquinone sulfonic acid sodium salt

## **2.6 Selecting Process Parameters**

Analysis of earlier results on cleaning efficiency had shown the cleaning solutions based on N-methyl pyrrolidone-water, nonionic surfactant Alarax BL-240 and citric acid catalyst are the most capable of removing LCA4/BA9 epoxy adhesive from stainless steel panels. Subsequently, the effect of process parameters, namely time, temperature, sonication and mechanical agitation was investigated. Steel coupons coated with LCA4/BA5 epoxy and cured for 6 hours at 200 F were used for this test. A statistically-designed grid of experiments was carried out using 50 C, 70 C, and 90 C as three levels of temperature. However, we did not find observable removal of epoxy at 50 C even after 48 hours and with mechanical agitation. Subsequently, the temperature was changed to 60 C, 75 C, and 90 C. Mechanical agitation was carried out using a shaker bath in which bottles containing coated coupons and cleaning solution were held firmly with clamps. Ultrasonic agitation was carried out using two ultrasonic cleaning baths. In one series of experiments, the bottles containing coupons and cleaning solution were exposed to ultrasonics. In a second series of experiments, the baths were filled with solvent and the coated coupons were exposed to ultrasonic vibration. The results showed that ultrasonic vibration has no appreciable effect

on the efficiency of the epoxy removal process. Temperature has a major effect on cleaning efficiency. Raising the temperature from 70 C to 90 C reduces the cleaning time from approximately 24 hours to 6 hours or less. The results of this study are given in Table 8.

### **2.7 Cleaning of Epoxy Adhesives** **FA8/BA5 and FA1/BA4**

Steel coupons were coated with both of these adhesives following the same procedure used for LCA4/BA5. The FA1/BA4 system presented some difficulty because it was too low in viscosity to coat the panel. The solution containing the epoxy and the activator was prereacted to increase the viscosity so it could be used to prepare a uniform coating. Experimentation demonstrated that heating for 10-15 minutes at 100 F is sufficient to increase the viscosity to the extent that it can be used to obtain a uniform coating on steel panels.

The coated samples were cured for 6 hours at 200 F. Differential calorimetric measurements confirmed that the adhesives were adequately cured having two Tg values, 84-85 C and 150 C.

The coupons were then cleaned using four cleaning solutions and two controls. The results are presented in Tables 9 and 10. Both residual weight and microscopic examination techniques were used for measuring cleaning efficiency. The results show that the cleaning solutions such as NMP/nonionic surfactant/B/citric acid, FA/cationic/A/citric acid and EL/nonionic/A which cleaned LCA4/BA5 are also effective for cleaning FA8/BA5 and FA1/BA4 adhesives. A, B, and C in these formulations are corrosion inhibitors discussed earlier. The solution NMP/nonionic/A/PTSA which was a moderately good solvent for LCA4/BA5 cleaned FA8/BA5 completely but was moderately successful in cleaning FA1/BA4 adhesive. Based on these results, it was concluded that the three cleaning solutions

**Table 8. Effect of Temperature and Agitation on Epoxy Removal Using NMP Solvent System<sup>(a)</sup>**

**(Steel coupons coated with LCA4/BA5 Epoxy Adhesive)**

Solution Number	Time, hours	Temperature, C	Agitation	Cleaning Efficiency		
				Average Residual Weight, percent <sup>(b)</sup>	Microscopic Evaluation <sup>(c)</sup>	Cleaning Efficiency <sup>(d)</sup>
56	24	90	Mechanical	0.005	7	Epoxy removed and dispersed into solution
57	4	90	ditto	77.160	97	Epoxy partially removed
58	24	60	"	29.265	49	Epoxy partially removed
59	4	60	"	102.522	160	Epoxy not removed
60	24	90	none	0.014	4	Epoxy removed and dispersed into solution
61	4	90	ditto	66.776	137	Epoxy partially removed
62	24	60	"	36.732	77	Epoxy partially removed
63	4	60	"	100.707	158	Epoxy not removed
64	16	75	"	0.000	5	Epoxy removed and dispersed into solution
65	16	75	"	0.000	6	ditto
66	16	75	"	0.000	2	"
67	16	75	sonication <sup>(e)</sup>	0.000	2	"
68	16	75	ditto	0.000	4	"
69	16	75	"	0.000	5	"

(a) NMP/nonionic/B/citric acid.

(b) Average of triplicate values.

(c) Average of points assigned to viewing area; point range 0 for no residue to 160 for total coverage.

(d) Epoxy removal or nonremoval after submersion in cleaning solution.

(e) 20 minute sonication with panel directly in cleaning solution.



**Table 9. Residual Weight and Cleaning Efficiency of Solvent Mixtures on FA8/BA5 Epoxy Cured at 6 Hours at 200 F**

Solution Number	Cleaning Solution <sup>(a)</sup>	Cleaning Efficiency		
		Average Residual Weight, percent <sup>(b)</sup>	Microscopic Evaluation <sup>(c)</sup>	Cleaning Efficiency <sup>(d)</sup>
31 Control	NMP/water	0.245	15	Complete removal, dispersed in solution
32 Control	EL/water	0.428	18	Complete removal with wiping
33 Control	FA/water	0.080	4	ditto
36	EL/nonionic/A	2.520	31	Complete removal with wiping on 1 panel, incomplete removal on 2
37	NMP/nonionic/A/PTSA	0.000	3	Complete removal, dispersed in solution
42	NMP/nonionic/B/citric acid	0.000	0	ditto
43	FA/cationic/A/citric acid	0.000	1.5	"

- (a) FA = furfuryl alcohol, NMP = N-methyl pyrrolidone, EL = ethyl lactate, PTSA = p-toluene sulfuric acid.
- (b) Average of better two (2) values of triplicates.
- (c) Points assigned to viewing area; point range 0 for no residue to 160 for total coverage.
- (d) Cleaned after 24 hours submersion in cleaning solution at 70 C.

**Table 10. Residual Weight and Cleaning Efficiency of Solvent Mixtures on FA1/BA4 Epoxy Cured at 6 Hours at 200 F**

Solution Number	Cleaning Solution <sup>(a)</sup>	Cleaning Efficiency		
		Average Residual Weight, percent <sup>(b)</sup>	Microscopic Evaluation <sup>(c)</sup>	Cleaning Efficiency <sup>(d)</sup>
28 Control	NMP/water	0.0000	17	Complete removal by wiping on 2 panels; incomplete removal on 1
29 Control	EL/water	0.034	20	Complete removal with wiping; visible spots
30 Control	FA/water	0.0000	1	Complete removal, dispersed in solution
34	EL/nonionic/A	0.005	17	Complete removal with wiping; visible spots
35	NMP/nonionic/A/PTSA	12.897	27	Incomplete removal on all 3 panels
44	NMP/nonionic/B/citric acid	0.000	6	Complete removal on 2 panels; incomplete on 1 <sup>(e)</sup>
45	FA/cationic/A/citric acid	0.000	3.5	Complete removal with wiping <sup>(e)</sup>

- (a) FA = furfuryl alcohol, NMP = N-methyl pyrrolidone, EL = ethyl lactate, PTSA = p-toluene sulfuric acid.
- (b) Average of better two (2) values of triplicates.
- (c) Points assigned to viewing area; point range 0 for no residue to 160 for total coverage.
- (d) After 24 hours submersion in cleaning solution at 70 C.
- (e) After 18 hours submersion in cleaning solution at 70 C.

are good cleaners of all the adhesive systems, namely LCA4/BA5, FA8/BA5 and FA1/BA4. However, the total data support the conclusion that the NMP water system is performing the best (shorter process time and complete removal).

### **3.0 Examine Compatibility With Substrate Metals**

#### **3.1 Selection and Procurement of Metal Substrates**

Six metal substrates, namely stainless steel 304, mu metal, chromium steel, beryllium, aluminum alloy Al 2017, and Ni-Fe alloy 4760 were procured for the compatibility study.

#### **3.2 Examine Corrosion and Pitting**

Three cleaning solutions were used to evaluate corrosion. The compositions of the solutions are given in Table 11. Hydrogen embrittlement and stress cracking experiments were not carried out because the potential of such occurrence was negligibly small.

**Table 11. Cleaning Solutions Used in Metal Compatibility Study**

Solvent	Weight, percent				
	Solvent	Water	Surfactant	Catalyst	Inhibitor
N-Methyl pyrrolidone	95.0	2.5	1.0 <sup>(a)</sup>	1.0 <sup>(c)</sup>	0.5 <sup>(d)</sup>
Ethyl lactate	83.5	15.0	1.0 <sup>(a)</sup>	--	0.5 <sup>(e)</sup>
Furfuryl alcohol	92.5	5.0	1.0 <sup>(b)</sup>	1.0 <sup>(c)</sup>	0.5 <sup>(e)</sup>

- (a) Nonionic, Antarox BL-240 (Rhone-Poulenc).
- (b) Cationic, Triton RW-50.
- (c) Citric acid monohydrate.
- (d) Anthraquinone 2-sulfonic acid, sodium salt monohydrate.
- (e) 5-Ethyl-2-methylpyridine.

The alloys were tested in the form of coupons (except beryllium) having nominal dimensions (in inches) of 2x0.75x0.125. A hole 3/16-inch in diameter was located at one end of the coupons to facilitate suspending them in the test solutions. Each alloy, except beryllium, was tested using triplicate coupons for each solution. Beryllium was tested in duplicates using parts of an accelerometer taken from a missile guidance system.

Prior to testing, the coupons were cleaned, weighed, and visually examined to determine the initial surface conditions. After preweighing, the alloys were tested by immersing the coupons continuously for 16 hours in the test solutions at a temperature of 75 C (167 F). The test solution was slowly agitated by magnetic stirrers during the test period.

After the test period, the coupons were removed from their solutions, rinsed with deionized water, dried, and reweighed. All of the coupons registered a weight loss. The results of the weight-loss measurements are shown in Figure 3 and presented in Table 12. Based on criteria established for a previous project\*, any weight loss less than 0.01 percent is considered acceptable for precision instruments. Additional compatibility criteria included no color change and pitting attack less than 0.5 mils.

### **3.3 Analysis of the Results**

Chromium steel was found to be incompatible with all the solutions after 16 hours of soaking when these additional criteria are used. In particular, chromium steel was stained after testing in furfuryl alcohol; and it was both stained and had pitting attack after testing in NMP and ethyl lactate. The other alloys did not exhibit any pitting attack or staining after their tests. The one beryllium coupon that did show a higher weight loss after testing in NMP did not show any visible sign of corrosion attack under a magnification of 200X. It is concluded that beryllium is compatible with NMP. It had been found in the previous study that alloys that were incompatible at 16 hour soaking were compatible at shorter soak times such as 1 hour. Therefore, it is quite likely that chromium steel would be compatible in the three test solutions at shorter soak times. Additional testing needs to be conducted to confirm this and recommend a schedule for components containing this alloy.

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\* AGMC/MAEL, Final Report on Metals-Detergent/Cleaner Compatibility Study, January 14, 1994.

# 16-hour Soak at 75 C (167 F)

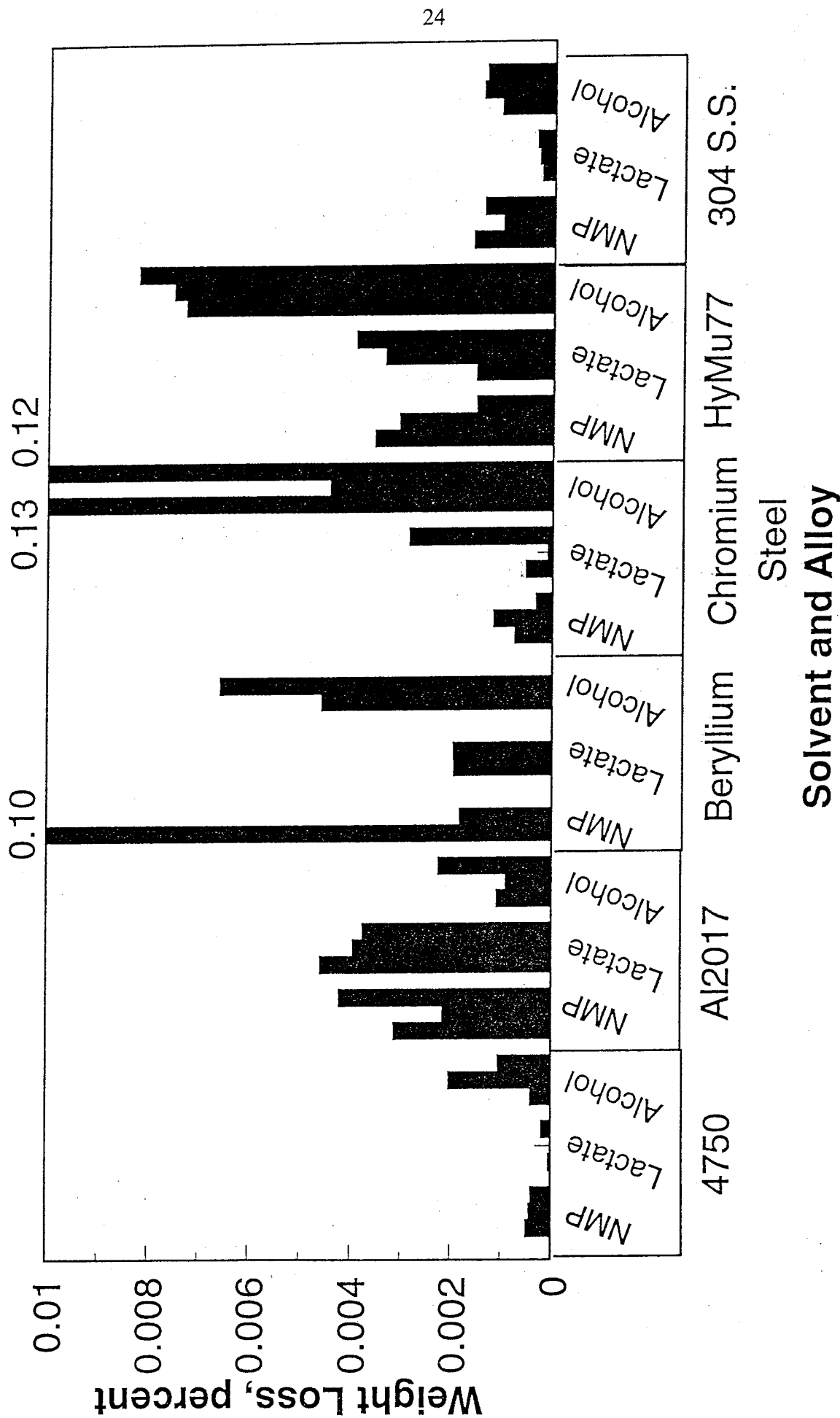


Figure 3. Weight-Loss Results of the Six Alloys Tested in the Three Test Solutions

Table 12. Weight Loss Results in Metal Compatibility Study

Coupon Alloy	No.	Solution	Initial Weight (g)	Final Weight (g)	Weight Loss (mg)	Weight Change (%)	Comments
4750	01	NMP	22.94887	22.94876	0.11	0.00048	Clean
4750	02	NMP	23.56765	23.56755	0.10	0.00042	Clean
4750	03	NMP	23.54984	23.54975	0.09	0.00038	Clean
4750	52	Ethyl Lactate	23.36791	23.36790	0.01	0.00004	Clean
4750	53	Ethyl Lactate	23.50500	23.50500	0.00	0.00000	Clean
4750	55	Ethyl Lactate	23.48176	23.48172	0.04	0.00017	Clean
4750	56	Furfuryl Alcohol	23.30002	23.29993	0.09	0.00039	Clean
4750	109	Furfuryl Alcohol	23.36251	23.36204	0.47	0.00201	Clean
4750	110	Furfuryl Alcohol	23.22443	23.22419	0.24	0.00103	Clean
Al2017	05	NMP	8.36598	8.36572	0.26	0.00311	Clean
Al2017	06	NMP	8.42521	8.42503	0.18	0.00214	Clean
Al2017	07	NMP	8.34272	8.34237	0.35	0.00420	Clean
Al2017	29	Ethyl Lactate	8.31741	8.31703	0.38	0.00457	Clean
Al2017	30	Ethyl Lactate	8.40129	8.40096	0.33	0.00393	Clean
Al2017	43	Ethyl Lactate	8.27941	8.27910	0.31	0.00374	Clean
Al2017	44	Furfuryl Alcohol	8.44196	8.44187	0.09	0.00107	Clean
Al2017	309	Furfuryl Alcohol	9.03914	9.03906	0.08	0.00089	Clean
Al2017	310	Furfuryl Alcohol	8.96425	8.96405	0.20	0.00223	Clean
Beryllium	34A	NMP	28.94926	28.91967	29.59	0.10221	Clean
Beryllium	34B	NMP	29.35639	29.35586	0.53	0.00181	Clean
Beryllium	35A	Ethyl Lactate	28.97875	28.97819	0.56	0.00193	Clean
Beryllium	35B	Ethyl Lactate	29.38216	29.38159	0.57	0.00194	Clean
Beryllium	36A	Furfuryl Alcohol	29.17400	29.17267	1.33	0.00456	Clean
Beryllium	36B	Furfuryl Alcohol	29.56664	29.56470	1.94	0.00656	Clean
Chromium Steel	01	NMP	23.23890	23.23873	0.17	0.00073	pits and stains
Chromium Steel	02	NMP	23.46425	23.46398	0.27	0.00115	pits and stains
Chromium Steel	07	NMP	22.29534	22.29527	0.07	0.00031	pits and stains
Chromium Steel	08	Ethyl Lactate	23.51778	23.51766	0.12	0.00051	pits and stains
Chromium Steel	22	Ethyl Lactate	23.31328	23.31326	0.02	0.00009	pits and stains
Chromium Steel	28	Ethyl Lactate	23.65290	23.65223	0.67	0.00283	pits and stains
Chromium Steel	29	Furfuryl Alcohol	21.51490	21.51197	2.93	0.01362	stains
Chromium Steel	65	Furfuryl Alcohol	23.13275	23.13173	1.02	0.00441	stains
Chromium Steel	66	Furfuryl Alcohol	23.44263	23.43973	2.90	0.01237	stains
HyMu77	01	NMP	6.52037	6.52014	0.23	0.00353	Clean
HyMu77	02	NMP	6.57400	6.57380	0.20	0.00304	Clean
HyMu77	03	NMP	6.66056	6.66046	0.10	0.00150	Clean
HyMu77	04	Ethyl Lactate	6.63060	6.63050	0.10	0.00151	Clean
HyMu77	05	Ethyl Lactate	6.60143	6.60121	0.22	0.00333	Clean
HyMu77	07	Ethyl Lactate	6.64186	6.64160	0.26	0.00391	Clean
HyMu77	08	Furfuryl Alcohol	6.59582	6.59534	0.48	0.00728	Clean
HyMu77	29	Furfuryl Alcohol	6.65259	6.65209	0.50	0.00752	Clean
HyMu77	30	Furfuryl Alcohol	6.57648	6.57594	0.54	0.00821	Clean
Type 304	01	NMP	20.84450	20.84417	0.33	0.00158	Clean
Type 304	02	NMP	21.26347	21.26326	0.21	0.00099	Clean
Type 304	03	NMP	21.28918	21.28889	0.29	0.00136	Clean
Type 304	07	Ethyl Lactate	21.35785	21.35780	0.05	0.00023	Clean
Type 304	08	Ethyl Lactate	21.31434	21.31428	0.06	0.00028	Clean
Type 304	28	Ethyl Lactate	21.32670	21.32663	0.07	0.00033	Clean
Type 304	58	Furfuryl Alcohol	21.41127	21.41105	0.22	0.00103	Clean
Type 304	59	Furfuryl Alcohol	20.92602	20.92573	0.29	0.00139	Clean
Type 304	60	Furfuryl Alcohol	21.14785	21.14757	0.28	0.00132	Clean

## **4.0 Optimize Cleaning System and Process**

The current cleaning solutions contain five different chemicals, namely solvent, water, surfactant, catalyst and inhibitor. The process parameters include time, temperature, and mechanical agitation. All these parameters could be optimized to provide the best cleaning solution and process.

### **4.1 Identify Key Material and Process Variables**

The results reported previously indicated that the key variables are:

- Percent water in NMP
- Percent catalyst
- Temperature
- Time.

In addition, the concentration of surfactant and corrosion inhibitor does not have a significant effect on cleaning efficiency. Sonication does not effect the cleaning results. Mechanical agitation improves the cleaning efficiency only marginally. Hence, the experimental grid was designed with only the four variables mentioned above. It was however, important to know the mid-point of these variables.

Preliminary experiments were carried out to determine the mid-point of the variables. Table 13 presents the effect of variations in the concentration of water, surfactant, and catalyst on cleaning efficiency. It was found that whereas 1 percent of water is sufficient to make N-methyl pyrrolidone effective, at least 10 percent of water is required for ethyl lactate to be effective as a cleaner. Hence, it was decided to have two separate experimental grids for optimization; one for the N-methyl pyrrolidone-based cleaning solution, and the other for the solution based on ethyl lactate. Higher catalyst concentration such as 4 percent was found to decrease cleaning efficiency. So the mid-point of catalyst concentration was chosen

**Table 13. Effect of Water, Surfactant, and Catalyst Concentration on Epoxy Removal of NMP Solvent System After 16 Hours at 75 C**

Solution Number	NMP, %	Water, %	Surfactant, %	Catalyst, %	Corrosion Inhibitor, %	Cleaning Efficiency		
						Average Residual Weight, % <sup>(a)</sup>	Microscopic Evaluation <sup>(b)</sup>	Cleaning Efficiency <sup>(c)</sup>
70	93.5	5	0	1	0.5	0.000	0	Epoxy removed and dispersed into solution
71	73.5	25	0	1	0.5	43.030	71	Epoxy partially removed, remaining not removed with wiping
72	91.5	5	2	1	0.5	0.000	3	Epoxy removed and dispersed into solution
73	71.5	25	2	1	0.5	70.743	100	Epoxy partially removed
74	90.5	5	0	4	0.5	4.077	17	Most of epoxy removed and dispersed into solution
75	70.5	25	0	4	0.5	102.037	150	Epoxy not removed
76	88.5	5	2	4	0.5	0.012	3	Epoxy removed and dispersed into solution
77	68.5	25	2	4	0.5	88.259	127	Epoxy loosened, and partially removed with wiping
78	81.0	15	1	2.5	0.5	36.634	83	ditto
79	81.0	15	1	2.5	0.5	9.895	29	Epoxy removed and dispersed in solution on two panels, partially removed on other
80	81.0	15	1	2.5	0.5	7.723	26	ditto
81	94.5	5	0	0	0.5	0.000	3	Epoxy removed and dispersed into solution
82	96.0	2.5	0	1	0.5	0.000	1	ditto
83	98.5	1.0	0	0	0.5	0.006	1	"

(a) Average of triplicate values.

(b) Average of points assigned to viewing area; point range 0 for no residue to 160 for total coverage.

(c) Epoxy removal or nonremoval after submersion in cleaning solution.



at 1.25 percent for N-methyl pyrrolidone based solution. For the ethyl lactate based solution, the acid catalyst was completely eliminated since it could potentially hydrolyze the solvent. The concentrations of the surfactant and corrosion inhibitor were kept constant at 1 and 0.5 percent, respectively.

#### **4.2 Optimization of Cleaning Process**

Optimization of the cleaning process was carried out in two separately designed statistical grids of cleaning experiments, one using N-methyl pyrrolidone and the other ethyl lactate. Stainless steel coupons coated with LCA4 and BA5 were used to test cleaning efficiency. The four parameters, namely percent of water, catalyst concentration, temperature, and time were varied. The results are presented in Tables 14 and 15.

The results obtained on N-methyl pyrrolidone indicate that time and temperature appear to have the most significant effect on cleaning efficiency.

At 90 C, 3 hours immersion gives incomplete cleaning. After 4 hours immersion, the residual epoxy is substantially reduced and becomes almost zero after 6 hours immersion. Water level at 8.75 percent works slightly better than at 2.5 percent. Catalyst concentration does not play a major role at this temperature. It can be varied between 0 and 2.5 percent.

At 75 C, the amount of residual epoxy was quite high after 3 hours immersion, but removal was complete after 8 hours of immersion with no catalyst. The best results are obtained when water level is maintained between 2.5 and 8.75 percent (preferably 8.75 percent) and no catalyst concentration at this temperature.

At 60 C, the panels did not show any significant cleaning after 7 hours. They had to be immersed overnight. After 16 hours, they all appeared to have no residual epoxy left and were perfectly clean.

**Table 14. Optimization of Epoxy Removal Process Using N-Methyl Pyrrolidone Based Cleaning Solutions**

Solu- tion No.	Solvent, %	Water, %	Catalyst, (a), %	Surfactant, (b), %	Inhibitor, (c) %	Temp., C	Time, hour	Average Residual <sup>(d)</sup> , %	Microscopic Evaluation <sup>(e)</sup>	Cleaning Efficiency <sup>(f)</sup>
91	88.5	8.75	1.25	1.0	0.5	90	4	1.604	8	Epoxy completely removed on 1 panel, almost complete on 2
92	89.75	8.75	0	1.0	0.5	90	6	0.000	3	Epoxy completely removed on all 3 panels
93	94.75	2.5	1.25	1.0	0.5	90	6	0.095	3	Epoxy completely removed on 1 panel, almost complete on 2
94	88.5	8.75	1.25	1.0	0.5	90	3	70.795	113	Epoxy partially removed on all 3 panels
95	87.25	8.75	2.5	1.0	0.5	90	3-6	0.000	0	Epoxy completely removed on all 3 panels
96	82.25	15.0	1.25	1.0	0.5	90	3-6	0.000	0	ditto
98	88.5	8.75	1.25	1.0	0.5	75	8	17.751	28	ditto
100	88.5	8.75	1.25	1.0	0.5	75	8	0.014	0	Epoxy completely removed on 2 panels; almost complete on 1
102	88.5	8.75	1.25	1.0	0.5	75	8	0.023	0	Epoxy completely removed on 2 panels; partially removed on 1
104	94.75	2.5	1.25	1.0	0.5	75	3	72.920	125	ditto
105	89.75	8.75	0	1.0	0.5	75	8	0.19	0	Epoxy completely removed on all 3 panels
107	89.75	8.75	0	1.0	0.5	75	3	98.292	160	Very little epoxy removed on all 3 panels
108	82.25	15.0	1.25	1.0	0.5	75	8	35.483	54	Epoxy almost completely removed on 1 panel; partially removed on 2

Table 14. (Continued)

Solu- tion No.	Solvent, %	Water, %	Catalyst, (a), %	Surfactant, (b), %	Inhibitor, (c) %	Temp., C	Time, hour	Average Residual <sup>(d)</sup> , %	Microscopic Evaluation <sup>(e)</sup>	Cleaning Efficiency <sup>(f)</sup>
109	94.75	2.5	1.25	1.0	0.5	75	8	0.026	1	Epoxy completely removed on 2 panels; partially removed on 1
110	82.25	15.0	1.25	1.0	0.5	75	3	87.737	125	Epoxy not removed on 2 panels; partially removed on 1
112	94.75	2.5	1.25	1.0	0.5	60	16 <sup>(g)</sup>	0.000	0	Epoxy completely removed on all 3 panels
113	87.25	8.75	2.5	1.0	0.5	60	16 <sup>(g)</sup>	0.000	0	"
114	89.75	8375	0	1.0	0.5	60	16 <sup>(g)</sup>	0.000	0	"
115	88.5	8.75	1.25	1.0	0.5	60	16 <sup>(g)</sup>	0.000	0	"
116	82.25	15.0	1.25	1.0	0.5	60	16 <sup>(g)</sup>	0.000	0	"
117	88.5	8.75	1.25	1.0	0.5	60	16 <sup>(g)</sup>	0.000	0	"

(a) Citric acid.

(b) Antarox BL-240 (nonionic).

(c) Anthraquinone-2-sulfonic acid, sodium salt monohydrate.

(d) Average of better (2) values.

(e) Average of points assigned to viewing area; 0 for no residue, to 160 for total coverage.

(f) Epoxy removal after submersion in cleaning solution.

(g) Until 7 hours, there was no apparent loss or lessening of epoxy adhesive from the panels at 60 C.

**Table 15. Optimization of Epoxy Removal Process Using Ethyl Lactate Based Cleaning Solution**

Solution No.	Solvent, %	Water, %	Surfactant <sup>(a)</sup> , %	Inhibitor <sup>(b)</sup> , %	Temp., C	Time, hr	Average Residual <sup>(c)</sup> , %	Microscopic Evaluation <sup>(d)</sup>	Cleaning Efficiency <sup>(e)</sup>
118	83.5	15	1.0	0.5	75	9.5	102.692	160	No epoxy removed on 2 panels; v. little on 1
119	78.5	20	1.0	0.5	75	23	58.547	75	Epoxy partially removed on all 3 panels
120	88.5	10	1.0	0.5	75	3	102.179	160	Epoxy not removed
121	78.5	20	1.0	0.5	75	3	102.173	160	ditto
122	83.5	15	1.0	0.5	75	9.5	77.113	106	Epoxy partially removed on 2 panels; v. little on 1
123	83.5	15	1.0	0.5	75	9.5	68.528	92	ditto
124	88.5	10	1.0	0.5	75	23	33.307	50	Epoxy partially removed on all 3 panels
125	83.5	15	1.0	0.5	60	16 <sup>(f)</sup>	0.000	0	Epoxy completely removed on 2 panels; partial on 1
126	88.5	10	1.0	0.5	60	16 <sup>(f)</sup>	11.665	18	Epoxy partially removed on all 3 panels
127	83.5	15	1.0	0.5	60	16 <sup>(f)</sup>	15.867	23	ditto
128	78.5	20	1.0	0.5	60	16 <sup>(f)</sup>	39.518	125	"
129	83.6	15	1.0	0.5	90	3	54.084	140	Epoxy partially removed on 1 panel; little removed on 2
130	88.5	10	1.0	0.5	90	6.5	18.653	25	Epoxy partially removed on all panels
131	78.5	20	1.0	0.5	90	6.5	42.512	58	ditto
132	83.5	15	1.0	0.5	90	6.5	11.698	16	"

(a) Antarox BL-240 (nonionic).

(b) 5-Ethyl-2-methyl pyridine.

(c) Average of better (2) values.

(d) Average of points assigned to viewing area; 0 for no residue, to 160 for total coverage.

(e) Epoxy removal or nonremoval after submersion in cleaning solution.

(f) There was no apparent loss of epoxy adhesive after 7 hours of immersion.

In general, it was concluded that NMP containing 8.75 percent water and 0 to 1.25 percent catalyst can clean the epoxy adhesive after 6 hours immersion at 90 C, after 8 hours at 75 C and after approximately 16 hours immersion at 60 C. Therefore, the time and temperature for cleaning can be varied depending on the production requirements. However, the cleaning processes recommended for 8 hours or less should use the NMP formulation at 90 C for 6 hours or at 75 C for 8 hours.

## **5.0 Confirmation With Actual Parts**

### **5.1 Procure Typical Parts**

Parts coated with a variety of epoxy adhesives were procured from AGMC. These included large and small parts, such as screws, nuts, etc.

### **5.2 Carry Out Cleaning and Measure Cleaning Efficiency**

Initial cleaning was carried out by soaking the parts in the N-methyl pyrrolidone based cleaning solution at 75 C for 8 hours. Epoxy removal was monitored by microscopic examination. The parts appeared perfectly clean.

## **6.0 Development of a Prototype Cleaning Process**

The results of the study were used to develop a prototype cleaning process to facilitate the work at AGMC. The prototype process documented the equipment, chemicals and the procedure required to remove epoxy adhesive from the electromechanical parts at AGMC along with quality assurance methods and solvent disposal procedure. The chemical composition of the solutions and process conditions were inferred from the results of optimization experiments. The document describing the prototype process is enclosed as Appendix A with this report.

## **7.0 Conclusions**

During the course of this program, Battelle has successfully developed a process for removing epoxy adhesive from delicate electromechanical devices repaired by AGMC. The process is based on the use of nontoxic, biodegradable water mixtures with N-methylpyrrolidone. The solubility parameters of this solvent were close to those of the cured epoxy resins used for adhesively joining the parts. The efficiency of epoxy removal increases significantly with increasing temperature and cycle time. Additives such as surfactants, epoxy degradation catalysts and corrosion protection agents can be used to improve process efficiency and safety. A thorough analysis of the effects of material and process parameters shows that:

- Addition of water to the three selected solvents is important for removing epoxy from the parts.
- The amount of water needed to be added to ethyl lactate is greater ( $\approx 15$  percent) compared to that needed for n-methyl pyrrolidone and furfuryl alcohol. This could be due to the lower solubility parameter of EL ( $\delta = 21.6 \text{ MPa}^{1/2}$  compared to those (22.9 and 24.3) for NMP and FA.
- Increasing temperature of the NMP solution reduces the time of soaking (immersion) of the parts.
- The addition of surfactants and acid catalysts does not have a significant effect on the epoxy removal process using NMP.
- The NMP-water process developed removes all candidate epoxies from steel substrates in 8 hours or less providing the soak temperature is 75 C or higher.

## **8.0 References**

1. Hearst, P. J., U.S. Navy, Washington, D.C., U.S. Statutory Invention Registration H300, July 7, 1987.

2. Nishibu, S. S., et al., Fuji Chemical Industrial Company, Ltd., Tokyo, Japan, U.S. Patent 4,444,858, April 24, 1984.
3. Beck Chemical Inc. [telephone (216) 941-8355], oral communication.
4. Miller Stevenson Co. [telephone (203) 743-3766], oral communication.
5. Dow Chemical [telephone (1-800) 258-CHEM], oral communication.
6. Bacon Industries [telephone (617) 926-2550], oral communication.
7. Kirk-Othmer, Encyclopedia of Chemical Technology, Vol. 7, pp. 135.

## **APPENDIX A**

### **PROTOTYPE CLEANING PROCESS**



## APPENDIX A

### PROTOTYPE CLEANING PROCESS

This process removes epoxy adhesives and coatings from electromechanical parts and components used in navigation and guidance equipments for the United States Air Force.

#### Materials and Equipment

##### Solvent System

The recommended process uses a N-methyl pyrrolidone (NMP) solvent-based system presented in Table A-1 which consists of solvent, water, a surfactant, a corrosion inhibitor, and a catalyst. The two major components are NMP solvent and water. The weight percentage of solvent in the preferred cleaner is 88.5 and the water level is 8.75 percent. The surfactant is maintained at 1 percent, corrosion inhibitor at 0.5 percent and the catalyst at 0 to 1.25 percent. The composition of the cleaning system is given in Table A-1.

Table A-1. Composition of Cleaning Solution		
Solvent	Function	Parts by Weight
N-methyl pyrrolidone	Solvent	88.5
Water	Co-Solvent	8.75
Antarox BL-240	Surfactant	1.0
Citric acid	Catalyst	0-1.25
5-Ethyl-2-methyl pyridine (EMP) or sodium salt of anthraquinone sulfonic acid (ASA)	Corrosion Inhibitor	0.5

## Equipment

The epoxy adhesives and coatings can be removed when exposed to the solvent-water system at 90 C for 6 hours or at 75 C for 8 hours. No agitation is required. The solvent is a high-boiling liquid. Its flash point is high (187 F). The fact that it contains water makes it less flammable and less explosive. Two types of equipment are recommended for the process — one for parts as large as 1000 cubic inches containing several liters of cleaning solution and one for small parts requiring only 50 to 60 ml of cleaning solution.

**Equipment #1:** A solvent bath, approximately 12 liters capacity, 29.2x32x12.2 cm in dimensions (VWR Standard bath Model #1203) or a bath of 33 liters capacity, 30.5x35.6x30.5 cm in dimensions (VWR Model #1295PC) can be used for the purpose. It should be provided with

- A cover fixed with a condenser with cooling water
- Sample platforms with holes (preferably stainless steel)
- Temperature control system
- Safety valve to release if pressure builds up.

The smaller bath may be used in case fewer parts need to be cleaned at a time. An impeller may be provided to circulate the solvent, thereby stopping build-up of epoxy debris near the parts. The bath temperature should be kept constant within  $\pm 1$  C. The larger equipment is suitable to remove epoxy adhesives from a large number of parts at a time or a large part.

**Equipment #2:** An air oven, approximately 13.9 cubic feet capacity provided with forced air circulation (VWR Model #1380 FM) may be used for this purpose. It should be provided with temperature control devices. Parts may be kept in small glass jars, above the platforms made from stainless steel screens (sheets with holes). The glass jars should be closed but not sealed so that pressure may not build up during solvent exposure.

## **Cleaning Procedure**

The recommended procedure consists of the following steps:

- Preparation of cleaning solution
- Exposure of parts to the cleaning solution
- Removal of parts from cleaning bath and rinsing
- Drying
- Microscopic examination
- Disposal of solvent waste.

## **Solution Preparation**

The compositions of the solvent system containing solvent, water, surfactant, catalyst and corrosion inhibitor are given in Table A-1.

Dissolve the additives, namely the surfactants, the catalyst and the corrosion inhibitor in water. In a vented hood, add the aqueous solution to the solvent and stir until all components dissolve, giving a homogeneous solution (clear).

## **Parts Exposure to Solvent System**

Place the parts to be cleaned on the stainless steel platform in a manner that the surface-to-volume ratio is maximum. This can be achieved by keeping them in a single layer without stacking and by keeping them separate from each other.

Increase the temperature of the solvent bath/air oven to 90 C. Introduce the platforms containing samples (Equipment #1) or the glass jars containing the samples (Equipment #2) into the hot solvent bath or air oven. Keep them at this temperature for approximately 6 hours. Maintain a uniform temperature,  $90 \pm 1$  during this operation.

## **Rinsing and Drying**

Remove the parts at the end of the exposure cycle and rinse them first with warm solvent followed by a wash with acetone. As an alternative to washing with acetone, give a second rinse with cold solvent followed by a spray rinse in distilled water. Allow the parts to dry at room temperature, preferably with forced clean air.

## **Quality Assurance**

Examine the surface of the parts with an optical microscope at 50 to 200 magnification to make sure that all the epoxy adhesive/coating has been removed. In case there is some residual epoxy on certain parts, expose these parts to hot solvent once more for 2 hours, rinse dry and inspect. Repeat until all epoxy residue is removed.

## **Solvent Disposal**

N-methyl pyrrolidone is an environmentally benign solvent and can be biodegraded into harmless byproducts. It would be possible to use the same solvent for at least 5 to 10 times, depending on the concentration of the debris. At the end, the solvent may be

- Recycled through distillation
- or ■ Disposed of in closed containers after filtering any remaining debris.

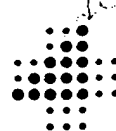
The debris will consist of epoxy adhesive and its degradation products and can be disposed of following procedures currently used at AGMC. MSDS are enclosed in Appendix B for all chemicals used.

## **APPENDIX B**

### **MSDS SHEETS FOR SOLVENTS AND ADDITIVES**

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B-1



DOCUMENT 6350

# American Burdick & Jackson

## Material Safety Data Sheet

MATERIAL SAFETY  
DATA SHEET

emergency telephone no. 312/973-3600 (American Scientific Products)  
chemtrec telephone no. 800/424-9300  
information telephone no. 616/726-3171 (American Burdick & Jackson)

### I. Identification

chemical name N-Methylpyrrolidone molecular weight 99.13  
chemical family Cyclic Amide formula C<sub>5</sub>H<sub>9</sub>NO  
synonyms NMP, N-Methyl-2-Pyrrolidone  
DOT proper shipping name Combustible Liquid, n.o.s.  
DOT hazard class Combustible Liquid  
DOT identification no. NA1993 CAS no. 872-50-4

N-METHYLPYRROLIDON

### II. Physical and Chemical Data

boiling point, 760mm Hg. 202°C freezing point -24.4°C evaporation rate (BuAc = 1) ca 0.1  
vapor pressure at 20°C < 1 mm Hg vapor density (air = 1) 3.4 solubility in water Complete  
% volatiles by volume ca 100 specific gravity (H<sub>2</sub>O = 1) @ 25°C 1.028 stability Stable  
hazardous polymerization Not expected to occur.  
appearance and odor Clear, colorless liquid with a slight amine odor.  
conditions to avoid Heat, sparks, open flame, open containers, and poor ventilation.

materials to avoid Strong oxidizing agents and strong acids.

hazardous decomposition products Incomplete combustion can generate carbon monoxide, nitrogen oxides, ammonia, and other toxic vapors.

### III. Fire and Explosion Hazard Data

flash point, (test method) 93°C (PM closed cup) auto ignition temperature 346°C  
flammable limits in air % by volume: lower limit 0.9 upper limit 3.9  
unusual fire and explosion hazards None, other than combustibility.

extinguishing media Carbon dioxide, dry chemical, or foam.

special fire fighting procedures Wear full protective clothing and self-contained breathing apparatus. Heat will build pressure and may rupture closed storage containers. Keep fire-exposed containers cool with water spray.

### IV. Hazardous Components

N-Methylpyrrolidone % ca 100 TLV not listed CAS no. 872-50-4

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American Burdick &amp; Jackson

Subsidiary of American  
Hospital Supply Corporation1953 South Harvey Street  
Muskegon MI 49442

## V. Health Hazards

Occupational Exposure Limits

OSHA	8-hour PEL	-	not listed
	Ceiling	-	not listed
	Peak	-	not listed

Concentration Immediately Dangerous to Health

OSHA/NIOSH	not listed
------------	------------

Odor Threshold

ACGIH	TLV-TWA	-	not listed
	TLV-STEL (15-min)	-	not listed

NSC & OHS	not listed
NIOSH	not listed

NIOSH	TLV-TWA	-	not listed
	TLV-C	-	not listed

Primary Routes of Entry

N-Methylpyrrolidone may exert its effects through inhalation, skin absorption, and ingestion.

Industrial Exposure: Route of Exposure/Signs and Symptoms

Inhalation: Exposure can cause slight irritation to mucous membranes.

Eye Contact: Liquid and high vapor concentration can be irritating. Direct contact may cause corneal damage.

Skin Contact: Prolonged or repeated skin contact can cause irritation.

Ingestion: Can cause gastrointestinal tract discomfort, diarrhea, and difficult breathing.

Effects of Overexposure

N-Methylpyrrolidone is an eye and slight mucous membrane irritant. Systemic effects information is inadequate/incomplete.

Medical Condition Aggravated by Exposure

Preclude from exposure those individuals susceptible to dermatitis.

Emergency First Aid

- Inhalation: Immediately remove to fresh air. If not breathing, administer mouth-to-mouth rescue breathing. If there is no pulse administer cardiopulmonary resuscitation (CPR). Contact physician immediately.
- Eye Contact: Rinse with copious amounts of water for at least 15 minutes. Get emergency medical assistance.
- Skin Contact: Flush thoroughly for at least 15 minutes. Wash affected skin with soap and water. Remove contaminated clothing and shoes. Wash clothing before re-use, and discard contaminated shoes. Get emergency medical assistance.
- Ingestion: Call local Poison Control Center for assistance. Contact physician immediately. Never induce vomiting or give anything by mouth to a victim unconscious or having convulsions.

**VI. Safety Measures and Equipment**

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- Ventilation: Adequate ventilation is required to protect personnel from exposure to chemical vapors and to minimize fire hazards. The choice of ventilation equipment, either local or general, will depend on the conditions of use, quantity of material, and other operating parameters.
- Respiratory: Use approved respirator equipment. Follow NIOSH and equipment manufacturer's recommendations to determine appropriate equipment (air-purifying, air-supplied, or self-contained breathing apparatus).
- Eyes: Safety glasses are considered minimum protection. Goggles or face shield may be necessary depending on quantity of material and conditions of use.
- Skin: Protective gloves and clothing are recommended. The choice of material must be based on chemical resistance and other user requirements. Generally, neoprene offers acceptable chemical resistance. Individuals who are acutely and specifically sensitive to N-methylpyrrolidone may require additional protective equipment.



**Storage:** N-Methylpyrrolidone should be protected from temperature extremes and direct sunlight. Proper storage of N-methylpyrrolidone must be determined based on other materials stored and their hazards and potential chemical incompatibility. In general, N-methylpyrrolidone should be stored in an acceptably protected and secure general storage room.

**Other:** Emergency eye wash fountains and safety showers should be available in the vicinity of any potential exposure

## VII. Spill and Disposal Data

**Spill Control:** Protect from ignition. Wear protective clothing and use approved respirator equipment. Absorb spilled material in an absorbent recommended for solvent spills and remove to a safe location for disposal by approved methods. If released to the environment, comply with all regulatory notification requirements.

**Waste Disposal:** Although N-methylpyrrolidone is not classified as an EPA hazardous waste, it should be disposed of in an environmentally acceptable manner.

Revision Date: 10/85

### KEY

ca	Approximately	STEL	Short Term Exposure Level
na	Not applicable	TLV	Threshold Limit Value
C	Ceiling	TWA	Time Weighted Average
PEL	Permissible Exposure Level	BuAc	Butyl Acetate
NSC	National Safety Council ("Fundamentals of Industrial Hygiene", 1983)		
OHS	Occupational Health Services ("Hazardline")		

OHS62443

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SECTION 1 CHEMICAL PRODUCTS & COMPANY IDENTIFICATION  
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OCCUPATIONAL HEALTH SERVICES, INC.  
11 WEST 42ND STREET, 12TH FLOOR  
NEW YORK, NEW YORK 10036  
1-800-445-MSDS (1-800-445-6737) OR  
1-212-789-3535

FOR EMERGENCY SOURCE INFORMATION  
CONTACT: 1-615-366-2000

CAS NUMBER: 687-47-8

SUBSTANCE: ETHYL (S)-(-)-LACTATE

## TRADE NAMES/SYNONYMS:

LACTIC ACID, ETHYL ESTER, (-)-; ETHYL-2-HYDROXYPROPIONATE;  
PROPANOIC ACID, 2-HYDROXY-, ETHYL ESTER, (S)-;  
(S)-ETHYL 2-HYDROXYPROPANOATE; 2-HYDROXYPROPANOIC ACID, ETHYL ESTER;  
ETHYL-L-LACTATE; ETHYL ALPHA-HYDROXYPROPIONATE; ETHYL 2-HYDROXYPROPANOATE;  
(-)-ETHYL LACTATE; ETHYL (S)-LACTATE; (S)-LACTIC ACID ETHYL ESTER;  
MICROPOSIT EC SOLVENT-11; UN 1192; STCC 4913165; C5H10O3; OHS62443

## CHEMICAL FAMILY:

ESTER, CARBOXYLIC, ALIPHATIC

CREATION DATE: 12/31/92

REVISION DATE: 03/24/93

-----  
SECTION 2 COMPOSITION/INFORMATION ON INGREDIENTS  
-----

COMPONENT : ETHYL (S)-(-)-LACTATE  
CAS NUMBER: 687-47-8  
PERCENTAGE: 100.0

OTHER CONTAMINANTS: NONE.

-----  
SECTION 3 HAZARDS IDENTIFICATION  
-----

CERCLA RATINGS (SCALE 0-3): HEALTH=U FIRE=2 REACTIVITY=0 PERSISTENCE=0  
NFPA RATINGS (SCALE 0-4): HEALTH=U FIRE=2 REACTIVITY=0

## EMERGENCY OVERVIEW:

CAUSES EYE IRRITATION. COMBUSTIBLE LIQUID AND VAPOR.  
KEEP AWAY FROM ALL IGNITION SOURCES. AVOID CONTACT WITH EYES, SKIN AND  
CLOTHING. WASH THOROUGHLY AFTER HANDLING.

## POTENTIAL HEALTH EFFECTS:

## INHALATION:

SHORT TERM EXPOSURE: MAY CAUSE DROWSINESS, DRUNKENESS AND PARALYSIS.

LONG TERM EFFECTS: NO INFORMATION IS AVAILABLE.

## SKIN CONTACT:

SHORT TERM EXPOSURE: MAY CAUSE IRRITATION.

LONG TERM EFFECTS: NO INFORMATION IS AVAILABLE.

EYE CONTACT:

SHORT TERM EXPOSURE: MAY CAUSE IRRITATION. ADDITIONAL EFFECTS MAY INCLUDE EYE DAMAGE.

LONG TERM EFFECTS: SAME EFFECTS AS SHORT TERM EXPOSURE.

INGESTION:

SHORT TERM EXPOSURE: MAY CAUSE DRUNKENNESS.

LONG TERM EFFECTS: NO INFORMATION IS AVAILABLE.

CARCINOGEN STATUS:

OSHA: N

NTP: N

IARC: N

---

SECTION 4

FIRST AID MEASURES

---

INHALATION:

FIRST AID- REMOVE FROM EXPOSURE AREA TO FRESH AIR IMMEDIATELY. IF BREATHING HAS STOPPED, PERFORM ARTIFICIAL RESPIRATION. KEEP PERSON WARM AND AT REST. TREAT SYMPTOMATICALLY AND SUPPORTIVELY. GET MEDICAL ATTENTION IMMEDIATELY.

SKIN CONTACT:

FIRST AID- REMOVE CONTAMINATED CLOTHING AND SHOES IMMEDIATELY. WASH AFFECTED AREA WITH SOAP OR MILD DETERGENT AND LARGE AMOUNTS OF WATER UNTIL NO EVIDENCE OF CHEMICAL REMAINS (APPROXIMATELY 15-20 MINUTES). GET MEDICAL ATTENTION IMMEDIATELY.

EYE CONTACT:

FIRST AID- WASH EYES IMMEDIATELY WITH LARGE AMOUNTS OF WATER OR NORMAL SALINE, OCCASIONALLY LIFTING UPPER AND LOWER LIDS, UNTIL NO EVIDENCE OF CHEMICAL REMAINS (APPROXIMATELY 15-20 MINUTES). GET MEDICAL ATTENTION IMMEDIATELY.

INGESTION:

FIRST AID- TREAT SYMPTOMATICALLY AND SUPPORTIVELY. GET MEDICAL ATTENTION IMMEDIATELY. IF VOMITING OCCURS, KEEP HEAD LOWER THAN HIPS TO PREVENT ASPIRATION.

NOTE TO PHYSICIAN

ANTIDOTE:

NO SPECIFIC ANTIDOTE. TREAT SYMPTOMATICALLY AND SUPPORTIVELY.

---

SECTION 5

FIRE FIGHTING MEASURES

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FIRE AND EXPLOSION HAZARD:

MODERATE FIRE HAZARD WHEN EXPOSED TO HEAT OR FLAME.

VAPORS ARE HEAVIER THAN AIR AND MAY TRAVEL A CONSIDERABLE DISTANCE TO A SOURCE OF IGNITION AND FLASH BACK.

VAPOR-AIR MIXTURES ARE EXPLOSIVE ABOVE FLASH POINT.

## EXTINGUISHING MEDIA:

DRY CHEMICAL, CARBON DIOXIDE, WATER SPRAY OR ALCOHOL-RESISTANT FOAM  
(1990 EMERGENCY RESPONSE GUIDEBOOK, DOT P 5800.5).

FOR LARGER FIRES, USE WATER SPRAY, FOG OR ALCOHOL-RESISTANT FOAM  
(1990 EMERGENCY RESPONSE GUIDEBOOK, DOT P 5800.5).

## ALCOHOL FOAM

(NFPA 325M, FIRE HAZARD PROPERTIES OF FLAMMABLE LIQUIDS, GASES, AND VOLATILE  
SOLIDS, 1991).

## FIREFIGHTING:

MOVE CONTAINER FROM FIRE AREA IF YOU CAN DO IT WITHOUT RISK. APPLY COOLING  
WATER TO SIDES OF CONTAINERS THAT ARE EXPOSED TO FLAMES UNTIL WELL AFTER FIRE  
IS OUT. STAY AWAY FROM ENDS OF TANKS. FOR MASSIVE FIRE IN CARGO AREA, USE  
UNMANNED HOSE HOLDER OR MONITOR NOZZLES; IF THIS IS IMPOSSIBLE, WITHDRAW FROM  
AREA AND LET FIRE BURN. WITHDRAW IMMEDIATELY IN CASE OF RISING SOUND FROM  
VENTING SAFETY DEVICE OR ANY DISCOLORATION OF TANK DUE TO FIRE. ISOLATE FOR  
1/2 MILE IN ALL DIRECTIONS IF TANK, RAIL CAR OR TANK TRUCK IS INVOLVED IN FIRE  
(1990 EMERGENCY RESPONSE GUIDEBOOK, DOT P 5800.5, GUIDE PAGE 26).

EXTINGUISH ONLY IF FLOW CAN BE STOPPED; USE WATER IN FLOODING AMOUNTS AS FOG,  
SOLID STREAMS MAY NOT BE EFFECTIVE. COOL CONTAINERS WITH FLOODING AMOUNTS OF  
WATER, APPLY FROM AS FAR A DISTANCE AS POSSIBLE. AVOID BREATHING VAPORS, KEEP  
UPWIND.

FLASH POINT: 115-120 F (46-49 C) (CC)

LOWER FLAMMABLE LIMIT: 1.5% @ 100 C

UPPER FLAMMABLE LIMIT: 11.4%

AUTOIGNITION: 752 F (400 C)

FLAMMABILITY CLASS(OSHA): II

## HAZARDOUS COMBUSTION PRODUCTS:

THERMAL DECOMPOSITION PRODUCTS MAY INCLUDE TOXIC OXIDES OF CARBON.

-----  
SECTION 6ACCIDENTAL RELEASE MEASURES  
-----

## OCCUPATIONAL SPILL:

SHUT OFF IGNITION SOURCES. STOP LEAK IF YOU CAN DO IT WITHOUT RISK. USE WATER  
SPRAY TO REDUCE VAPORS. FOR SMALL SPILLS, TAKE UP WITH SAND OR OTHER  
ABSORBENT MATERIAL AND PLACE INTO CONTAINERS FOR LATER DISPOSAL. FOR LARGER  
SPILLS, DIKE FAR AHEAD OF SPILL FOR LATER DISPOSAL. NO SMOKING, FLAMES OR  
FLARES IN HAZARD AREA. KEEP UNNECESSARY PEOPLE AWAY; ISOLATE HAZARD AREA AND  
DENY ENTRY.

-----  
SECTION 7HANDLING AND STORAGE  
-----

OBSERVE ALL FEDERAL, STATE AND LOCAL REGULATIONS WHEN STORING THIS SUBSTANCE.

STORE IN ACCORDANCE WITH 29 CFR 1910.106.

BONDING AND GROUNDING: SUBSTANCES WITH LOW ELECTROCONDUCTIVITY, WHICH

MAY BE IGNITED BY ELECTROSTATIC SPARKS, SHOULD BE STORED IN CONTAINERS WHICH MEET THE BONDING AND GROUNDING GUIDELINES SPECIFIED IN NFPA 77-1983, RECOMMENDED PRACTICE ON STATIC ELECTRICITY.

STORE IN A TIGHTLY CLOSED CONTAINER.

STORE IN A COOL, DRY PLACE PROTECTED AGAINST LIGHT.

STORE BETWEEN 50-70 F.

STORE AWAY FROM INCOMPATIBLE SUBSTANCES.

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SECTION 8            EXPOSURE CONTROLS/PERSONAL PROTECTION

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EXPOSURE LIMITS:

NO OCCUPATIONAL EXPOSURE LIMITS ESTABLISHED BY OSHA, ACGIH, OR NIOSH.

VENTILATION:

PROVIDE LOCAL EXHAUST OR GENERAL DILUTION VENTILATION. VENTILATION EQUIPMENT MUST BE EXPLOSION-PROOF.

EYE PROTECTION:

EMPLOYEE MUST WEAR SPLASH-PROOF OR DUST-RESISTANT SAFETY GOGGLES TO PREVENT EYE CONTACT WITH THIS SUBSTANCE.

EMERGENCY EYE WASH: WHERE THERE IS ANY POSSIBILITY THAT AN EMPLOYEE'S EYES MAY BE EXPOSED TO THIS SUBSTANCE, THE EMPLOYER SHOULD PROVIDE AN EYE WASH FOUNTAIN WITHIN THE IMMEDIATE WORK AREA FOR EMERGENCY USE.

CLOTHING:

EMPLOYEE MUST WEAR APPROPRIATE PROTECTIVE (IMPERVIOUS) CLOTHING AND EQUIPMENT TO PREVENT REPEATED OR PROLONGED SKIN CONTACT WITH THIS SUBSTANCE.

GLOVES:

EMPLOYEE MUST WEAR APPROPRIATE PROTECTIVE GLOVES TO PREVENT CONTACT WITH THIS SUBSTANCE.

RESPIRATOR:

THE FOLLOWING RESPIRATORS ARE RECOMMENDED BASED ON INFORMATION FOUND IN THE PHYSICAL DATA, TOXICITY AND HEALTH EFFECTS SECTIONS. THEY ARE RANKED IN ORDER FROM MINIMUM TO MAXIMUM RESPIRATORY PROTECTION. THE SPECIFIC RESPIRATOR SELECTED MUST BE BASED ON CONTAMINATION LEVELS FOUND IN THE WORK PLACE, MUST BE BASED ON THE SPECIFIC OPERATION, MUST NOT EXCEED THE WORKING LIMITS OF THE RESPIRATOR AND MUST BE JOINTLY APPROVED BY THE NATIONAL INSTITUTE FOR OCCUPATIONAL SAFETY AND HEALTH AND THE MINE SAFETY AND HEALTH ADMINISTRATION (NIOSH-MSHA).

ANY CHEMICAL CARTRIDGE RESPIRATOR WITH ORGANIC VAPOR CARTRIDGE(S) AND A FULL FACEPIECE.

ANY GAS MASK WITH ORGANIC VAPOR CANISTER (CHIN-STYLE OR FRONT- OR BACK-MOUNTED CANISTER), WITH A FULL FACEPIECE.

ANY TYPE 'C' SUPPLIED-AIR RESPIRATOR WITH A FULL FACEPIECE OPERATED IN PRESSURE-DEMAND OR OTHER POSITIVE PRESSURE MODE OR WITH A FULL FACEPIECE, HELMET OR HOOD OPERATED IN A CONTINUOUS-FLOW MODE.

ANY SELF-CONTAINED BREATHING APPARATUS WITH A FULL FACEPIECE OPERATED IN PRESSURE-DEMAND OR OTHER POSITIVE PRESSURE MODE.

FOR FIREFIGHTING AND OTHER IMMEDIATELY DANGEROUS TO LIFE OR HEALTH CONDITIONS:

ANY SELF-CONTAINED BREATHING APPARATUS THAT HAS A FULL FACEPIECE AND IS OPERATED IN A PRESSURE-DEMAND OR OTHER POSITIVE-PRESSURE MODE.

ANY SUPPLIED-AIR RESPIRATOR THAT HAS A FULL FACEPIECE AND IS OPERATED IN A PRESSURE-DEMAND OR OTHER POSITIVE-PRESSURE MODE IN COMBINATION WITH AN AUXILIARY SELF-CONTAINED BREATHING APPARATUS OPERATED IN PRESSURE-DEMAND OR OTHER POSITIVE-PRESSURE MODE.

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## SECTION 9 PHYSICAL AND CHEMICAL PROPERTIES

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DESCRIPTION: COLORLESS LIQUID WITH A MILD CHARACTERISTIC ODOR.

MOLECULAR WEIGHT: 118.15

MOLECULAR FORMULA: C-H3-C-H-(O-H)-C-O2-C2-H5

BOILING POINT: 309 F (154 C)

MELTING POINT: -13 TO -15 F (-25 TO -26 C)

VAPOR DENSITY: 4.1

SPECIFIC GRAVITY: 1.042

WATER SOLUBILITY: MISCIBLE, PARTIALLY DECOMPOSES

VOLATILITY: 100%

ODOR THRESHOLD: 14 PPM

EVAPORATION RATE: (ETHER=1) >1

SOLVENT SOLUBILITY: SOLUBLE IN ALCOHOLS, ETHER, KETONES, ESTERS, CHLOROFORM, GASOLINE, HYDROCARBONS, OILS.

REFRACTIVE INDEX: 1.4130

BULK DENSITY: 8.55 LBS/GAL AT 20 C

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## SECTION 10 STABILITY AND REACTIVITY

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REACTIVITY:

STABLE UNDER NORMAL TEMPERATURES AND PRESSURES.

CONDITIONS TO AVOID:

AVOID CONTACT WITH HEAT, SPARKS, FLAMES, OR OTHER SOURCES OF IGNITION. VAPORS MAY BE EXPLOSIVE AND POISONOUS; DO NOT ALLOW UNNECESSARY PERSONNEL IN AREA.

DO NOT OVERHEAT CONTAINERS; CONTAINERS MAY VIOLENTLY RUPTURE AND TRAVEL A CONSIDERABLE DISTANCE IN HEAT OF FIRE.

INCOMPATIBILITIES:

ETHYL LACTATE:

OXIDIZERS (STRONG): FIRE AND EXPLOSION HAZARD.

HAZARDOUS DECOMPOSITION:  
THERMAL DECOMPOSITION PRODUCTS MAY INCLUDE TOXIC OXIDES OF CARBON.

POLYMERIZATION:  
HAZARDOUS POLYMERIZATION HAS NOT BEEN REPORTED TO OCCUR UNDER NORMAL TEMPERATURES AND PRESSURES.

-----  
SECTION 11TOXICOLOGY INFORMATION  
-----

ETHYL (S)-(-)-LACTATE:  
CARCINOGEN STATUS: NONE.  
LOCAL EFFECTS: IRRITANT- EYE.  
ACUTE TOXICITY LEVEL: NO DATA AVAILABLE.  
TARGET EFFECTS: POISONING MAY AFFECT THE CENTRAL NERVOUS SYSTEM.

## HEALTH EFFECTS

## INHALATION:

## ETHYL LACTATE:

ACUTE EXPOSURE- AT VERY HIGH CONCENTRATIONS MAY CAUSE CENTRAL NERVOUS SYSTEM DEPRESSION WITH DROWSINESS, NARCOSIS, AND DEATH DUE TO PARALYSIS OF RESPIRATORY SYSTEM.  
CHRONIC EXPOSURE- NO DATA AVAILABLE.

## SKIN CONTACT:

## ETHYL LACTATE:

ACUTE EXPOSURE- MAY CAUSE IRRITATION. ONE CASE OF SENSITIZATION HAS BEEN REPORTED.  
CHRONIC EXPOSURE- NO DATA AVAILABLE.

## EYE CONTACT:

## ETHYL LACTATE:

## IRRITANT.

ACUTE EXPOSURE- MAY CAUSE IRRITATION, POSSIBLY SEVERE. PERMANENT EYE DAMAGE HAS BEEN REPORTED IN RABBITS.  
CHRONIC EXPOSURE- REPEATED AND PROLONGED CONTACT WITH IRRITANTS MAY CAUSE CONJUNCTIVITIS.

## INGESTION:

## ETHYL LACTATE:

ACUTE EXPOSURE- MAY CAUSE NARCOSIS. CENTRAL NERVOUS SYSTEM EFFECTS HAVE BEEN REPORTED IN MICE.  
CHRONIC EXPOSURE- NO DATA AVAILABLE.

-----  
SECTION 12ECOLOGICAL INFORMATION  
-----

ENVIRONMENTAL IMPACT RATING (0-4): NO DATA AVAILABLE

ACUTE AQUATIC TOXICITY: NO DATA AVAILABLE

DEGRADABILITY: NO DATA AVAILABLE

LOG BIOCONCENTRATION FACTOR (BCF): NO DATA AVAILABLE

LOG OCTANOL/WATER PARTITION COEFFICIENT: NO DATA AVAILABLE

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SECTION 13DISPOSAL INFORMATION

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OBSERVE ALL FEDERAL, STATE AND LOCAL REGULATIONS WHEN DISPOSING OF THIS SUBSTANCE.

DISPOSAL MUST BE IN ACCORDANCE WITH STANDARDS APPLICABLE TO GENERATORS OF HAZARDOUS WASTE, 40 CFR 262. EPA HAZARDOUS WASTE NUMBER D001.  
100 POUND CERCLA SECTION 103 REPORTABLE QUANTITY.

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SECTION 14TRANSPORTATION INFORMATION

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DEPARTMENT OF TRANSPORTATION HAZARD CLASSIFICATION 49-CFR 172.101:  
COMBUSTIBLE LIQUID

DEPARTMENT OF TRANSPORTATION LABELING REQUIREMENTS 49-CFR 172.101 AND  
SUBPART E:  
NONE

DEPARTMENT OF TRANSPORTATION PACKAGING REQUIREMENTS: NONE  
EXCEPTIONS: 49-CFR 173.118A

FINAL RULE ON HAZARDOUS MATERIALS REGULATIONS (HMR, 49 CFR PARTS 171-180),  
DOCKET NUMBERS HM-181, HM-181A, HM-181B, HM-181C, HM-181D AND HM-204.  
EFFECTIVE DATE OCTOBER 1, 1991. HOWEVER, COMPLIANCE WITH THE REGULATIONS IS  
AUTHORIZED ON AND AFTER JANUARY 1, 1991. (55 FR 52402, 12/21/90)

EXCEPT FOR EXPLOSIVES, INHALATION HAZARDS, AND INFECTIOUS SUBSTANCES, THE  
EFFECTIVE DATE FOR HAZARD COMMUNICATION REQUIREMENTS IS EXTENDED TO  
OCTOBER 1, 1993. (56 FR 47158, 09/18/91)

U.S. DEPARTMENT OF TRANSPORTATION SHIPPING NAME-ID NUMBER, 49 CFR 172.101:  
ETHYL LACTATE-UN 1192

U.S. DEPARTMENT OF TRANSPORTATION HAZARD CLASS OR DIVISION, 49 CFR 172.101:  
3 - FLAMMABLE LIQUID

U.S. DEPARTMENT OF TRANSPORTATION PACKING GROUP, 49 CFR 172.101:  
PG III

U.S. DEPARTMENT OF TRANSPORTATION LABELING REQUIREMENTS, 49 CFR 172.101  
AND SUBPART E:  
FLAMMABLE LIQUID

U.S. DEPARTMENT OF TRANSPORTATION PACKAGING AUTHORIZATIONS:  
EXCEPTIONS: 49 CFR 173.150  
NON-BULK PACKAGING: 49 CFR 173.203  
BULK PACKAGING: 49 CFR 173.242

U.S. DEPARTMENT OF TRANSPORTATION QUANTITY LIMITATIONS 49 CFR 172.101:



PASSENGER AIRCRAFT OR RAILCAR: 60 L  
CARGO AIRCRAFT ONLY: 220 L

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SECTION 15REGULATORY INFORMATION  
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TSCA STATUS: N

CERCLA SECTION 103 (40CFR302.4): N  
SARA SECTION 302 (40CFR355.30): N  
SARA SECTION 304 (40CFR355.40): N  
SARA SECTION 313 (40CFR372.65): N  
OSHA PROCESS SAFETY (29CFR1910.119): N  
CALIFORNIA PROPOSITION 65: N

SARA HAZARD CATEGORIES, SARA SECTIONS 311/312 (40 CFR 370.21)

ACUTE HAZARD: Y  
CHRONIC HAZARD: N  
FIRE HAZARD: Y  
REACTIVITY HAZARD: N  
SUDDEN RELEASE HAZARD: N

-----  
SECTION 16OTHER  
-----

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SURFACTANTS &amp; SPECIALTIES

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24 HOUR EMERGENCY (800) 334-7577

MEDICAL EMERGENCY (800) 228-5635

CHEMICAL EMERGENCY (SPILL, LEAK, FIRE)

CHEMTREC (800) 424-9300

05

## I - IDENTIFICATION

RESOURCE NO. 3302 REV. 5 DATE 08/92 ANTAROX BL-240 SURFACTANT

TRADE NAME: ANTAROX BL-240 SURFACTANT

EXPORT TRADE NAME: ANTAROX BL-240 SURFACTANT

CAS REGISTRY NO: 00068603-25-8

CAS REGISTRY NAME: ALCOHOLS, C8-10, ETHOXYLATED PROPOXYLATED

SYNONYM: MIXED OCTYL/DECYL ALCOHOLS, ETHOXYLATED PROPOXYLATED

MOLECULAR FORMULA: C H O

31 64 10

MOLECULAR WEIGHT: 582.00

05

## II - PHYSICAL & HEALTH HAZARD INGREDIENTS

CAS REGISTRY NO.: 00000075-21-8

CAS REGISTRY NAME: OXIRANE

COMMON NAME: ETHYLENE OXIDE (EO)

HAZARD % : LESS THAN 0.0002

HAZARD REFERENCE: PEL(TLV) TWA: 1 PPM; ACTION LEVEL: 0.5 PPM  
SEE SEC. 07-OCCUPAT'L CONTROL PROCEDURES.  
NOT SUBJECT TO REPORTING UNDER SARA  
SECTION 313 AT THIS CONCENTRATION.

05

## III - PHYSICAL & CHEMICAL CHARACTERISTICS

BOILING POINT:	NO DATA FOUND
VAPOR PRESSURE:	NOT VOLATILE
VAPOR DENSITY (AIR=1):	NOT VOLATILE
WATER SOLUBILITY:	SOLUBLE
MELTING/FREEZING POINT:	NO DATA FOUND
APPEARANCE:	CLEAR TO SLIGHTLY HAZY FREE FLOWING LIQUID
SPECIFIC GRAV. (WATER=1):	0.9870
PERCENTAGE VOLATILES:	0.50 % WATER-MAX
EVAPORATION RATE:	NOT VOLATILE
PH OF SOLUTION:	6.00 - 8.00 10% SOLUTION
ODOR:	BLAND

05

## IV - PHYSICAL HAZARD DATA

FLASH POINT:	255.00 DEG F (OC)
AUTOIGNITION TEMP.:	NO DATA FOUND
FLAMMABLE LIMITS:	NO DATA FOUND

FIRE FIGHTING MEDIA:  
USE MEDIA PROPER TO PRIMARY CAUSE OF FIRE.SPECIAL FIRE FIGHTING PROCEDURES:  
NONE KNOWN

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CHEMICAL EMERGENCY (SPILL, LEAK, FIRE)  
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ANTAROX BL-240 SURFACTANT

05 IV - PHYSICAL HAZARD DATA (CONT.)

FIRE/EXPLOSION HAZARDS:  
NONE KNOWN

NFPA HAZARD CODES - HEALTH/FLAMMABILITY/REACTIVITY 1 1 0

HMS HAZARD CODES - HEALTH/FLAMMABILITY/REACTIVITY 2 1 0

05 V - REACTIVITY DATA

HAZARDOUS POLYMERIZATION: STABILITY: STABLE  
WILL NOT OCCUR

CONDITIONS TO AVOID:  
NONE KNOWN

INCOMPATIBLE MATERIALS:  
STRONG OXIDIZING OR REDUCING AGENTS.

HAZARDOUS DECOMPOSITION PRODUCTS:  
NONE KNOWN

05 VI - HEALTH HAZARD DATA

- ACUTE TOXICITY -

ORAL TOXICITY:  
RAT LD50: 2400 MG/KG

DERMAL TOXICITY:  
NO DATA FOUND.

INHALATION TOXICITY:  
NO DATA FOUND.

SKIN IRRITATION:  
RABBIT; PRIMARY IRRITATION INDEX: 0.83; SLIGHT IRRITANT.

EYE IRRITATION:  
RABBIT; MODERATE OCULAR IRRITANT

SENSITIZER: NO

DOT CORROSIVE: NO

PRIMARY ROUTE(S) OF ENTRY: CONTACT

- SIGNS & SYMPTOMS OF EXPOSURE -

SYMPTOMS OF INGESTION:  
NO EFFECTS OF EXPOSURE EXPECTED.

SYMPTOMS OF INHALATION:  
IF MISTED, WILL CAUSE IRRITATION OF MUCOUS MEMBRANES, NOSE, EYES AND THROAT. COUGHING, DIFFICULTY IN BREATHING.



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## VI - HEALTH HAZARD DATA (CONT.)

## SYMPTOMS OF SKIN CONTACT:

NO EFFECTS OF EXPOSURE EXPECTED DUE TO CONTACT. MAY POSSIBLY CAUSE IRRITATION OR DERMATITIS IN SOME INDIVIDUALS UPON PROLONGED CONTACT.

## SYMPTOMS OF EYE CONTACT:

WILL CAUSE PAINFUL BURNING OR STINGING OF EYES AND LIDS, WATERING OF EYES, AND INFLAMMATION OF CONJUNCTIVA.

## - EMERGENCY FIRST AID PROCEDURES -

## FIRST AID FOR INGESTION:

GENERAL PRECAUTIONARY MEASURES SUGGEST INDUCING VOMITING IMMEDIATELY BY GIVING TWO GLASSES OF WATER AND STICKING FINGER DOWN THROAT. NEVER GIVE ANYTHING BY MOUTH TO AN UNCONSCIOUS PERSON. CALL A PHYSICIAN.

## FIRST AID FOR INHALATION:

REMOVE TO FRESH AIR. IF NOT BREATHING, GIVE ARTIFICIAL RESPIRATION, PREFERABLY MOUTH-TO-MOUTH. IF BREATHING IS DIFFICULT, GIVE OXYGEN. CALL A PHYSICIAN.

## FIRST AID FOR SKIN CONTACT:

FOR ALL FOREIGN MATERIALS, WASH AFTER EXPOSURE.

## FIRST AID FOR EYE CONTACT:

IMMEDIATELY FLUSH EYES WITH PLENTY OF WATER FOR AT LEAST 15 MINUTES. CALL A PHYSICIAN.

## MEDICAL CONDITIONS AGGRAVATED BY EXPOSURE:

NO DATA FOUND.

## EXPOSURE LIMITS:

SEE SECTION 02 FOR PHYSICAL & HEALTH HAZARD INGREDIENTS.

## - OTHER TOXICITY -

OTHER ACUTE TOXICITY: NO DATA FOUND

SUB CHRONIC ORAL TOXICITY: NO DATA FOUND

SUB CHRONIC INHALATION TOXICITY: NO DATA FOUND

SUB CHRONIC MISCELLANEOUS TOXICITY: NO DATA FOUND

AQUATIC TOXICITY: NO DATA FOUND

## TUMORIGENICITY:

THE NTP LISTS ETHYLENE OXIDE IN THE "FOURTH ANNUAL REPORT ON CARCINOGENS," REFERENCING AN IARC REPORT PUBLISHED IN 1984. THIS REPORT STATES THAT THERE IS SUFFICIENT EVIDENCE FOR CARCINOGENICITY TO EXPERIMENTAL ANIMALS. HOWEVER, WHEN PRODUCT IS HANDLED IN ACCORDANCE WITH SECTION 07, "OCCUPATIONAL CONTROL PROCEDURES," NEITHER ACUTE NOR LONG TERM HAZARD FROM ETHYLENE OXIDE IS EXPECTED.

MUTAGENICITY: NO DATA FOUND

REPRODUCTIVE TOXICITY: NO DATA FOUND

MISCELLANEOUS TOXICITY: NO DATA FOUND

05

## VII - OCCUPATIONAL CONTROL PROCEDURES

## VENTILATION:

TRACES OF ETHYLENE OXIDE (EO) COULD ACCUMULATE IN HEADSPACE OF STORAGE/TRANSPORT VESSELS. USE WITH ADEQUATE VENTILATION, ESPECIALLY WHERE DRUMS OR TANKWAGONS ARE BEING OPENED.



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ANTAROX BL-240 SURFACTANT

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## VII - OCCUPATIONAL CONTROL PROCEDURES (CONT.)

### RESPIRATORY PROTECTION:

USE NIOSH-APPROVED RESPIRATOR, IF ADEQUATE VENTILATION CANNOT BE PROVIDED AT ANY TIME, OR IF THERE IS A POSSIBILITY OF EXCESSIVE CONTACT WITH HEADSPACE ABOVE THE DRUM OR TANKWAGON.

### EYE PROTECTION:

CHEMICAL GOGGLES.

### SKIN PROTECTION:

NONE REQUIRED.

### PERSONAL HYGIENE:

WASH THOROUGHLY AFTER HANDLING.

### PROTECTIVE MEASURES DURING REPAIR/MAINTENANCE OF EQUIPMENT:

WASH EQUIPMENT THOROUGHLY WITH STEAM OR WARM WATER UNTIL CLEAN. CHECK FOR FLAMMABLES WITH AN "EXPLOSION METER" AND ALSO CHECK THE OXYGEN LEVEL WITH AN OXYGEN METER. IN ALL CASES, FOLLOW GOOD INDUSTRIAL SAFETY PRACTICES BEFORE ENTERING EQUIPMENT.

05

## VIII - PRECAUTIONS FOR SAFE HANDLING, STORAGE, AND USE

### PRECAUTIONARY MEASURES:

AVOID CONTACT WITH EYES. WASH THOROUGHLY AFTER HANDLING. AVOID BREATHING MIST. USE ADEQUATE VENTILATION FOR MISTING OPERATIONS. EO, A CANCER AND REPRODUCTIVE HAZARD, MAY BE PRESENT IN THIS PRODUCT. REPEATED EXPOSURE MAY BE HARMFUL. IF CONTROL PROCEDURES IN SECTION 07 ARE FOLLOWED, AIR SPACE CONCENTRATIONS SHOULD BE BELOW THE OSHA ESTABLISHED "ACTION LEVEL".

### SPILL/LEAK CLEAN-UP PROCEDURES:

ABSORB WITH EARTH, SAND OR SIMILAR INERT MATERIAL AND DISPOSE OF WITH SOLID WASTE ACCORDING TO FEDERAL, STATE AND LOCAL REGULATIONS. FLUSH SPILL AREA WITH WATER.

### DISPOSAL METHOD:

DISPOSE OF WITH LIQUID WASTE ACCORDING TO FEDERAL, STATE AND LOCAL REGULATIONS.

EPA HAZARDOUS SUBSTANCE REPORTABLE QUANTITY: NOT LISTED

RCRA CLASS: NOT REGULATED

05

## IX - TRANSPORTATION DATA

### - DOMESTIC DATA -

DOT SHIPPING NAME: NOT REGULATED

DOT HAZARD CLASS:

HAZARDOUS INGREDIENT(S): NONE

UN NUMBER: NONE

### - EXPORT DATA -

EXPORT SHIPPING NAME: NOT REGULATED

EXPORT HAZARD CLASS:

HAZARDOUS INGREDIENT(S): NONE

UN NUMBER: NONE

### PREPARED BY:

Health, Safety and Environmental Affairs

Effective Date: 08/14/92

The information herein is given in good faith  
but no warranty is expressed nor implied.

05



END OF MATERIAL SAFETY DATA SHEET  
RHÔNE-POULENC

OHS14480

---

SECTION 1 CHEMICAL PRODUCTS & COMPANY IDENTIFICATION

---

OCCUPATIONAL HEALTH SERVICES, INC.  
11 WEST 42ND STREET, 12TH FLOOR  
NEW YORK, NEW YORK 10036  
1-800-445-MSDS (1-800-445-6737) OR  
1-212-789-3535

FOR EMERGENCY SOURCE INFORMATION  
CONTACT: 1-615-366-2000

CAS NUMBER: 104-90-5  
RTECS NUMBER: TJ6825000

SUBSTANCE: METHYL ETHYLPYRIDINE

## TRADE NAMES/SYNONYMS:

5-ETHYL-2-PICOLINE; 5-ETHYL-2-METHYLPYRIDINE; MED; ALDEHYDINE;  
3-ETHYL-6-METHYLPYRIDINE; 5-ETHYL-ALPHA-PICOLINE; 2-METHYL-5-ETHYLPYRIDINE;  
ALDEHYDECOLLIDINE; 6-METHYL-3-ETHYLPYRIDINE; STCC 4935660; UN 2300; OHS14480

## CHEMICAL FAMILY:

Aldehyde, aliphatic

CREATION DATE: 03/04/85

REVISION DATE: 08/26/93

---

SECTION 2 COMPOSITION/INFORMATION ON INGREDIENTS

---

COMPONENT : METHYL ETHYLPYRIDINE  
CAS NUMBER: 104-90-5  
PERCENTAGE: 100

OTHER CONTAMINANTS: NONE

---

SECTION 3 HAZARDS IDENTIFICATION

---

CERCLA RATINGS (SCALE 0-3): HEALTH=3 FIRE=2 REACTIVITY=0 PERSISTENCE=1  
NFPA RATINGS (SCALE 0-4): HEALTH=3 FIRE=2 REACTIVITY=0

## EMERGENCY OVERVIEW:

METHYL ETHYLPYRIDINE IS A COLORLESS LIQUID WITH A STRONG ODOR.

Harmful if absorbed through skin. Causes respiratory tract, skin and eye burns. Combustible liquid and vapor.

Do not breathe vapor or mist. Do not get in eyes, on skin, or on clothing.

Keep away from all ignition sources. Keep container tightly closed. Wash thoroughly after handling. Use only with adequate ventilation.

## POTENTIAL HEALTH EFFECTS:

## INHALATION:

SHORT TERM EFFECTS: May cause irritation, possibly severe. Additional effects may include vomiting and chest pain.

LONG TERM EFFECTS: Same effects as short term exposure.

**SKIN CONTACT:**

SHORT TERM EFFECTS: May cause irritation, possibly severe. May also cause death.

LONG TERM EFFECTS: Same effects as short term exposure.

**EYE CONTACT:**

SHORT TERM EFFECTS: May cause irritation, possibly severe.

LONG TERM EFFECTS: Same effects as short term exposure.

**INGESTION:**

SHORT TERM EFFECTS: May cause gastrointestinal irritation. Additional effects may include lack of appetite, nausea, vomiting, headache, weakness and drunkenness.

LONG TERM EFFECTS: No information is available.

**CARCINOGEN STATUS:**

OSHA: N

NTP: N

IARC: N

---

**SECTION 4****FIRST AID MEASURES**

---

**INHALATION:**

FIRST AID- Remove from exposure area to fresh air immediately. If breathing has stopped, give artificial respiration. Maintain airway and blood pressure and administer oxygen if available. Keep affected person warm and at rest. Treat symptomatically and supportively. Administration of oxygen should be performed by qualified personnel. Get medical attention immediately.

**SKIN CONTACT:**

FIRST AID- Remove contaminated clothing and shoes immediately. Wash affected area with soap or mild detergent and large amounts of water until no evidence of chemical remains (at least 15-20 minutes). In case of chemical burns, cover area with sterile, dry dressing. Bandage securely, but not too tightly. Get medical attention immediately.

**EYE CONTACT:**

FIRST AID- Wash eyes immediately with large amounts of water, occasionally lifting upper and lower lids, until no evidence of chemical remains (at least 15-20 minutes). Continue irrigating with normal saline until the pH has returned to normal (30-60 minutes). Cover with sterile bandages. Get medical attention immediately.

**INGESTION:**

FIRST AID- Treat symptomatically and supportively. Get medical attention and advice immediately on whether to use gastric lavage. Gastric lavage performed by qualified medical personnel may be suitable if there are no signs of perforation.

**NOTE TO PHYSICIAN****ANTIDOTE:**

No specific antidote. Treat symptomatically and supportively.

---

SECTION 5FIRE FIGHTING MEASURES

---

## FIRE AND EXPLOSION HAZARD:

Moderate fire hazard when exposed to heat or flame.

Vapors are heavier than air and may travel a considerable distance to a source of ignition and flash back.

Vapor-air mixtures are explosive above flash point.

## EXTINGUISHING MEDIA:

Dry chemical, carbon dioxide, water spray or regular foam (1990 Emergency Response Guidebook, DOT P 5800.5).

For larger fires, use water spray, fog or regular foam (1990 Emergency Response Guidebook, DOT P 5800.5).

## Alcohol foam

(NFPA 325m, fire hazard properties of flammable liquids, gases, and volatile solids, 1991).

## FIREFIGHTING:

Move container from fire area if you can do it without risk. Apply cooling water to sides of containers that are exposed to flames until well after fire is out. Stay away from ends of tanks (1990 Emergency Response Guidebook, DOT P 5800.5, Guide Page 60).

Extinguish only if flow can be stopped; use water in flooding amounts as fog. Cool containers with flooding quantities of water, apply from as far a distance as possible. Avoid breathing toxic vapors, keep upwind.

Fire fighting phases: Use water spray, dry chemical, alcohol foam, or carbon dioxide. Use water spray to disperse vapors and to protect men attempting to stop a leak. Water spray may be used to flush spills away from exposures (NFPA 49, Hazardous Chemicals Data, 1975).

FLASH POINT: 155 F (68 C) (OC)

LOWER FLAMMABLE LIMIT: 1.1%

UPPER FLAMMABLE LIMIT: 6.6%

AUTOIGNITION: 939 F (504 C)

FLAMMABILITY CLASS(OSHA): IIIA

## HAZARDOUS COMBUSTION PRODUCTS:

Thermal decomposition may release toxic and/or hazardous gases.

---

SECTION 6ACCIDENTAL RELEASE MEASURES

---

## OCCUPATIONAL SPILL:

Do not touch spilled material. Stop leak if you can do it without risk. For small spills, take up with sand or other absorbent material and place into containers for later disposal. For small dry spills, with clean shovel place material into clean, dry container and cover. Move containers from spill



area. For larger spills, dike far ahead of spill for later disposal. Keep unnecessary people away. Isolate hazard area and deny entry.

---

SECTION 7HANDLING AND STORAGE

---

Observe all federal, state and local regulations when storing this substance.

Storage: Protect against physical damage. Isolate from powerful oxidizing materials. Store in a cool, dry, well-ventilated location, away from any area where the fire hazard may be acute. Outside or detached storage is preferred. Do not use copper or copper alloy storage vessels (NFPA 49, Hazardous Chemicals Data, 1975).

---

SECTION 8EXPOSURE CONTROLS/PERSONAL PROTECTION

---

## EXPOSURE LIMITS:

No occupational exposure limits established by OSHA, ACGIH, or NIOSH.

## VENTILATION:

Provide local exhaust or process enclosure ventilation system.

## EYE PROTECTION:

Employee must wear splash-proof or dust-resistant safety goggles and a faceshield to prevent contact with this substance.

## Emergency wash facilities:

Where there is any possibility that an employee's eyes and/or skin may be exposed to this substance, the employer should provide an eye wash fountain and quick drench shower within the immediate work area for emergency use.

## CLOTHING:

Employee must wear appropriate protective (impervious) clothing and equipment to prevent any possibility of skin contact with this substance.

## GLOVES:

Employee must wear appropriate protective gloves to prevent contact with this substance.

## RESPIRATOR:

The following respirators are recommended based on information found in the physical data, toxicity and health effects sections. They are ranked in order from minimum to maximum respiratory protection.

The specific respirator selected must be based on contamination levels found in the work place, must be based on the specific operation, must not exceed the working limits of the respirator and must be jointly approved by the National Institute for Occupational Safety and Health and the Mine Safety and Health Administration (NIOSH-MSHA).

Any chemical cartridge respirator with an organic vapor cartridge(s) with an acid gas cartridge(s) and a full facepiece.

Any gas mask with organic vapor canister (chin-style or front- or

back-mounted canister), with a full facepiece, providing protection against acid gases.

Any type 'C' supplied-air respirator with a full facepiece operated in pressure-demand or other positive pressure mode or with a full facepiece, helmet or hood operated in continuous-flow mode.

Any self-contained breathing apparatus with a full facepiece operated in pressure-demand or other positive pressure mode.

FOR FIREFIGHTING AND OTHER IMMEDIATELY DANGEROUS TO LIFE OR HEALTH CONDITIONS:

Any self-contained breathing apparatus that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode.

Any supplied-air respirator that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode in combination with an auxiliary self-contained breathing apparatus operated in pressure-demand or other positive-pressure mode.

---

SECTION 9 PHYSICAL AND CHEMICAL PROPERTIES

---

DESCRIPTION: Colorless liquid with a sharp penetrating odor.

MOLECULAR WEIGHT: 121.18

MOLECULAR FORMULA: C<sub>8</sub>H<sub>11</sub>N

BOILING POINT: 352 F (178 C)

MELTING POINT: -95 F (-70 C)

VAPOR DENSITY: 4.2

SPECIFIC GRAVITY: 0.919

WATER SOLUBILITY: slightly soluble

SOLVENT SOLUBILITY: Alcohol, benzene, concentrated sulfuric acid, ether, acids

---

SECTION 10 STABILITY AND REACTIVITY

---

REACTIVITY:

Stable under normal temperatures and pressures.

CONDITIONS TO AVOID:

Avoid contact with heat, sparks, flames, or other sources of ignition. Vapors may be explosive. Avoid overheating of containers; containers may violently rupture in heat of fire. Avoid contamination of water sources.

INCOMPATIBILITIES:

METHYL ETHYLPYRIDINE:

OXIDIZERS: Incompatible.

NITRIC ACID: Explosion hazard.

HAZARDOUS DECOMPOSITION:

Thermal decomposition may release toxic and/or hazardous gases.

POLYMERIZATION:

Hazardous polymerization has not been reported to occur under normal temperatures and pressures.

---

SECTION 11TOXICOLOGY INFORMATION

---

## METHYL ETHYLPYRIDINE:

10 mg/24 hours open skin-rabbit severe irritation; 500 mg open skin-rabbit severe irritation; 250 ug open eye-rabbit severe irritation; 368 mg/kg oral-rabbit LD50; 1000 ppm/4 hours inhalation-rat LCLo; 826 mg/kg subcutaneous-rat LD50; 282 mg/kg oral-mouse LD50; 1000 mg/kg skin-rabbit LD50; CARCINOGEN STATUS: None.

Methyl ethylpyridine is a severe eye, skin and mucous membrane irritant and is toxic.

## HEALTH EFFECTS

## INHALATION:

## METHYL ETHYLPYRIDINE:

## CORROSIVE.

ACUTE EXPOSURE- Inhalation of vapors may cause severe irritation to the respiratory tract, vomiting and chest discomfort. A moderate dose was lethal to rats tested.

CHRONIC EXPOSURE- Repeated or prolonged exposure may cause mucous membrane irritation.

## SKIN CONTACT:

## METHYL ETHYLPYRIDINE:

## CORROSIVE/TOXIC.

ACUTE EXPOSURE- Contact with the skin may cause severe irritation with burns and necrosis. A relatively low dose was lethal to rabbits tested but systemic effects were not reported.

CHRONIC EXPOSURE- Repeated or prolonged exposure may cause dermatitis.

## EYE CONTACT:

## METHYL ETHYLPYRIDINE:

## CORROSIVE.

ACUTE EXPOSURE- Contact with the eyes may cause severe irritation with burns. When applied to rabbit eyes, the substance was rated 9 on a scale of 1-10 on irritancy. 0.5 ml of solution has been reported to cause severe burns.

CHRONIC EXPOSURE- Repeated or prolonged exposure may cause conjunctivitis.

## INGESTION:

## METHYL ETHYLPYRIDINE:

## CORROSIVE/TOXIC.

ACUTE EXPOSURE- Ingestion may cause severe irritation of the mouth and stomach. A relatively low dose was lethal to rabbits tested but systemic effects were not reported. Pyridines, in general, may cause central nervous system depression with headache, dizziness, nausea and vomiting. Anorexia, fatigue, and mental depression may also occur.

CHRONIC EXPOSURE- No data available.

---

SECTION 12ECOLOGICAL INFORMATION

---

-----  
ENVIRONMENTAL IMPACT RATING (0-4): no data available

ACUTE AQUATIC TOXICITY: no data available

DEGRADABILITY: no data available

LOG BIOCONCENTRATION FACTOR (BCF): no data available

LOG OCTANOL/WATER PARTITION COEFFICIENT: no data available  
-----

SECTION 13

DISPOSAL INFORMATION

-----  
Observe all federal, state and local regulations when disposing of this substance.  
-----

SECTION 14

TRANSPORTATION INFORMATION

-----  
DEPARTMENT OF TRANSPORTATION HAZARD CLASSIFICATION 49-CFR 172.101:  
Corrosive material

Department of Transportation labeling requirements 49-CFR 172.101 and  
SUBPART E:  
Corrosive

DEPARTMENT OF TRANSPORTATION PACKAGING REQUIREMENTS: 49-CFR 173.245  
EXCEPTIONS: 49-CFR 173.244

Final rule on hazardous materials regulations (HMR, 49 CFR parts 171-180),  
docket numbers HM-181, HM-181a, HM-181b, HM-181c, HM-181d and HM-204.  
Effective date October 1, 1991. However, compliance with the regulations is  
authorized on and after January 1, 1991. (55 FR 52402, 12/21/90)

Except for explosives, inhalation hazards, and infectious substances, the  
effective date for hazard communication requirements is extended to  
October 1, 1993. (56 FR 47158, 09/18/91)

U.S. DEPARTMENT OF TRANSPORTATION SHIPPING NAME-ID NUMBER, 49 CFR 172.101:  
2-methyl-5-ethylpyridine-UN 2300

U.S. DEPARTMENT OF TRANSPORTATION HAZARD CLASS OR DIVISION, 49 CFR 172.101:  
6.1 - Poisonous materials

U.S. DEPARTMENT OF TRANSPORTATION PACKING GROUP, 49 CFR 172.101:  
PG III

U.S. DEPARTMENT OF TRANSPORTATION LABELING REQUIREMENTS, 49 CFR 172.101  
AND SUBPART E:  
Keep away from food

U.S. DEPARTMENT OF TRANSPORTATION PACKAGING AUTHORIZATIONS:  
EXCEPTIONS: 49 CFR 173.153



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 ATTN: SAFETY DIRECTOR  
 BATTELLE MEMORIAL INST  
 ATTN VERA ERAC  
 COLUMBUS OH

 DATE: 03/18/94  
 CUST#: 130451

## MATERIAL SAFETY DATA SHEET PAGE 1

## SECTION 1. - - - - - CHEMICAL IDENTIFICATION - - - - -

 PRODUCT #: 12324-2 NAME: ANTHRAQUINONE-2-SULFONIC ACID, SODIUM SALT  
 MONOHYDRATE, 97%

## SECTION 2. - - - - - COMPOSITION/INFORMATION ON INGREDIENTS - - - - -

 CAS #: 131-08-8  
 MF: C14H8O5S

## SYNONYMS

 9,10-ANTHRAQUINONE-2-SODIUM SULFONATE \* 2-ANTHRAQUINONESULFONATE  
 SODIUM \* ANTHRAQUINONE-2-SULFONATE SODIUM SALT \* 2-  
 ANTHRAQUINONESULFONIC ACID SODIUM SALT \* SILVER SALT \* SODIUM-2-  
 ANTHRACHINONESULFONATE \* SODIUM BETA-ANTHRAQUINONESULFONATE \* SODIUM  
 2-ANTHRAQUINONESULFONATE \* SODIUM 9,10-ANTHRAQUINONE-2-SULFONATE \* 2-  
 SULFOANTHRAQUINONE SODIUM SALT \*

## SECTION 3. - - - - - HAZARDS IDENTIFICATION - - - - -

## LABEL PRECAUTIONARY STATEMENTS

 IRRITANT  
 IRRITATING TO EYES, RESPIRATORY SYSTEM AND SKIN.  
 IN CASE OF CONTACT WITH EYES, RINSE IMMEDIATELY WITH PLENTY OF  
 WATER AND SEEK MEDICAL ADVICE.  
 WEAR SUITABLE GLOVES AND EYE/FACE PROTECTION.

## SECTION 4. - - - - - FIRST-AID MEASURES - - - - -

 IN CASE OF CONTACT, IMMEDIATELY FLUSH EYES WITH COPIOUS AMOUNTS OF  
 WATER FOR AT LEAST 15 MINUTES.  
 IN CASE OF CONTACT, IMMEDIATELY WASH SKIN WITH SOAP AND COPIOUS  
 AMOUNTS OF WATER.  
 IF INHALED, REMOVE TO FRESH AIR. IF NOT BREATHING GIVE ARTIFICIAL  
 RESPIRATION. IF BREATHING IS DIFFICULT, GIVE OXYGEN.  
 IF SWALLOWED, WASH OUT MOUTH WITH WATER PROVIDED PERSON IS CONSCIOUS.  
 CALL A PHYSICIAN.  
 WASH CONTAMINATED CLOTHING BEFORE REUSE.

## SECTION 5. - - - - - FIRE FIGHTING MEASURES - - - - -

## EXTINGUISHING MEDIA

 WATER SPRAY.  
 CARBON DIOXIDE, DRY CHEMICAL POWDER OR APPROPRIATE FOAM.

## SPECIAL FIREFIGHTING PROCEDURES

 WEAR SELF-CONTAINED BREATHING APPARATUS AND PROTECTIVE CLOTHING TO  
 PREVENT CONTACT WITH SKIN AND EYES.

## UNUSUAL FIRE AND EXPLOSIONS HAZARDS

EMITS TOXIC FUMES UNDER FIRE CONDITIONS.

## SECTION 6. - - - - - ACCIDENTAL RELEASE MEASURES - - - - -

 WEAR RESPIRATOR, CHEMICAL SAFETY GOGGLES, RUBBER BOOTS AND HEAVY  
 RUBBER GLOVES.  
 SWEEP UP, PLACE IN A BAG AND HOLD FOR WASTE DISPOSAL.  
 AVOID RAISING DUST.

CONTINUED ON NEXT PAGE

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\*\* TOTAL PAGE.003 \*\*



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**M A T E R I A L   S A F E T Y   D A T A   S H E E T**

**PAGE 2**

DATE: 03/18/94  
CUST#: 130451

PRODUCT #: 12324-2      NAME: ANTHRAQUINONE-2-SULFONIC ACID, SODIUM SALT MONOHYDRATE, 97%

VENTILATE AREA AND WASH SPILL SITE AFTER MATERIAL PICKUP IS COMPLETE.

SECTION 7. - - - - - HANDLING AND STORAGE - - - - -

REFER TO SECTION 8.

SECTION 8. - - - - - EXPOSURE CONTROLS/PERSONAL PROTECTION - - - - -

CHEMICAL SAFETY GOGGLES.  
RUBBER GLOVES.  
NIOSH/MSHA-APPROVED RESPIRATOR.  
SAFETY SHOWER AND EYE BATH.  
MECHANICAL EXHAUST REQUIRED.  
DO NOT BREATHE DUST.  
DO NOT GET IN EYES, ON SKIN, ON CLOTHING.  
WASH THOROUGHLY AFTER HANDLING.  
IRRITANT.  
KEEP TIGHTLY CLOSED.  
STORE IN A COOL DRY PLACE.

SECTION 9. - - - - - PHYSICAL AND CHEMICAL PROPERTIES - - - - -

APPEARANCE AND ODOR  
LIGHT-YELLOW POWDER  
MELTING POINT: 300 °C

SECTION 10. - - - - - STABILITY AND REACTIVITY - - - - -

INCOMPATIBILITIES  
STRONG OXIDIZING AGENTS  
HAZARDOUS COMBUSTION OR DECOMPOSITION PRODUCTS  
TOXIC FUMES OF:  
CARBON MONOXIDE, CARBON DIOXIDE  
SULFUR OXIDES

SECTION 11. - - - - - TOXICOLOGICAL INFORMATION - - - - -

ACUTE EFFECTS  
MAY BE HARMFUL BY INHALATION, INGESTION, OR SKIN ABSORPTION.  
CAUSES EYE AND SKIN IRRITATION.  
MATERIAL IS IRRITATING TO MUCOUS MEMBRANES AND UPPER  
RESPIRATORY TRACT.  
TO THE BEST OF OUR KNOWLEDGE, THE CHEMICAL, PHYSICAL, AND  
TOXICOLOGICAL PROPERTIES HAVE NOT BEEN THOROUGHLY INVESTIGATED.

TECS NO: CB1095550  
2-ANTHRACENESULFONIC ACID, 9,10-DIHYDRO-9,10-DIOXO-, SODIUM SALT  
TOXICITY DATA

CONTINUED ON NEXT PAGE

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## MATERIAL SAFETY DATA SHEET

PAGE 3

 DATE: 03/18/94  
 CUST#: 130451

PRODUCT #: 12324-2

NAME: ANTHRAQUINONE-2-SULFONIC ACID, SODIUM SALT  
MONOHYDRATE, 97%

IPR-RAT LD50: 730 MG/KG

GISAAA 55(4), 93, 90

IPR-MUS LD50: 630 MG/KG

GISAAA 55(4), 93, 90

JRL-GPG LD50: 21 GM/KG

GISAAA 45(3), 73, 80

 ONLY SELECTED REGISTRY OF TOXIC EFFECTS OF CHEMICAL SUBSTANCES  
 (RTECS) DATA IS PRESENTED HERE. SEE ACTUAL ENTRY IN RTECS FOR  
 COMPLETE INFORMATION.

## SECTION 12. ----- ECOLOGICAL INFORMATION -----

DATA NOT YET AVAILABLE.

## SECTION 13. ----- DISPOSAL CONSIDERATIONS -----

 DISSOLVE OR MIX THE MATERIAL WITH A COMBUSTIBLE SOLVENT AND BURN IN A  
 CHEMICAL INCINERATOR EQUIPPED WITH AN AFTERBURNER AND SCRUBBER.  
 OBSERVE ALL FEDERAL, STATE AND LOCAL ENVIRONMENTAL REGULATIONS.

## SECTION 14. ----- TRANSPORT INFORMATION -----

CONTACT ALDRICH CHEMICAL COMPANY FOR TRANSPORTATION INFORMATION.

## SECTION 15. ----- REGULATORY INFORMATION -----

REVIEWS, STANDARDS, AND REGULATIONS

NOHS 1974: HZD 81978; NIS 1; TNF 28; NOS 2; TNE 700

NOES 1983: HZD 81978; NIS 3; TNF 113; NOS 5; TNE 1799; TFE 806

EPA TSCA CHEMICAL INVENTORY, JUNE 1993

## SECTION 16. ----- OTHER INFORMATION -----

 THE ABOVE INFORMATION IS BELIEVED TO BE CORRECT BUT DOES NOT PURPORT TO  
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OHS84211

---

SECTION 1 CHEMICAL PRODUCTS & COMPANY IDENTIFICATION

---

OCCUPATIONAL HEALTH SERVICES, INC.  
11 WEST 42ND STREET, 12TH FLOOR  
NEW YORK, NEW YORK 10036  
1-800-445-MSDS (1-800-445-6737) OR  
1-212-789-3535

FOR EMERGENCY SOURCE INFORMATION  
CONTACT: 1-615-366-2000

CAS NUMBER: 5949-29-1  
RTECS NUMBER: GE7810000

SUBSTANCE: HYDROUS CITRIC ACID

## TRADE NAMES/SYNONYMS:

1,2,3-PROPANETRICARBOXYLIC ACID, 2-HYDROXY-, MONOHYDRATE;  
2-HYDROXY-1,2,3-PROPANETRICARBOXYLIC ACID MONOHYDRATE;  
CITRIC ACID, MONOHYDRATE; CITRIC ACID MONOHYDRATE;  
NEUTRALIZER FOR ALKALINES (MALLINCKRODT); C6H10O8; OHS84211

## CHEMICAL FAMILY:

Carboxylic acid, aliphatic

CREATION DATE: 09/01/87

REVISION DATE: 03/24/93

---

SECTION 2 COMPOSITION/INFORMATION ON INGREDIENTS

---

COMPONENT : HYDROUS CITRIC ACID  
CAS NUMBER: 5949-29-1  
PERCENTAGE: 100.0

OTHER CONTAMINANTS: NONE

---

SECTION 3 HAZARDS IDENTIFICATION

---

CERCLA RATINGS (SCALE 0-3): HEALTH=3 FIRE=1 REACTIVITY=0 PERSISTENCE=0  
NFPA RATINGS (SCALE 0-4): HEALTH=3 FIRE=1 REACTIVITY=0

## EMERGENCY OVERVIEW:

HYDROUS CITRIC ACID IS AN ODORLESS, COLORLESS SOLID.

Causes eye burns. Causes respiratory tract and skin irritation. May form flammable or explosive dust-air mixtures.

Do not get in eyes, on skin, or on clothing. Keep away from all ignition sources. Avoid breathing dust. Keep container tightly closed. Avoid dispersion of dust. Wash thoroughly after handling. Use only with adequate ventilation.

## POTENTIAL HEALTH EFFECTS:

## INHALATION:

SHORT TERM EFFECTS: May cause irritation. Additional effects may include coughing and shortness of breath.



LONG TERM EFFECTS: No information available on significant adverse effects.

SKIN CONTACT:

SHORT TERM EFFECTS: May cause irritation.

LONG TERM EFFECTS: Same effects as short term exposure.

EYE CONTACT:

SHORT TERM EFFECTS: May cause irritation, possibly severe. Additional effects may include sores.

LONG TERM EFFECTS: Same effects as short term exposure.

INGESTION:

SHORT TERM EFFECTS: May cause sore throat, vomiting and digestive disorders.

LONG TERM EFFECTS: May cause tooth decay.

CARCINOGEN STATUS:

OSHA: N

NTP: N

IARC: N

---

SECTION 4

FIRST AID MEASURES

---

INHALATION:

FIRST AID- Remove from exposure area to fresh air immediately. If breathing has stopped, perform artificial respiration. Keep person warm and at rest. Treat symptomatically and supportively. Get medical attention immediately.

SKIN CONTACT:

FIRST AID- Remove contaminated clothing and shoes immediately. Wash affected area with soap or mild detergent and large amounts of water until no evidence of chemical remains (approximately 15-20 minutes). Get medical attention immediately.

EYE CONTACT:

FIRST AID- Wash eyes immediately with large amounts of water, occasionally lifting upper and lower lids, until no evidence of chemical remains (at least 15-20 minutes). Continue irrigating with normal saline until the pH has returned to normal (30-60 minutes). Cover with sterile bandages. Get medical attention immediately.

INGESTION:

FIRST AID- Treat symptomatically and supportively. Get medical attention immediately. If vomiting occurs, keep head lower than hips to prevent aspiration.

NOTE TO PHYSICIAN

ANTIDOTE:

No specific antidote. Treat symptomatically and supportively.

---

SECTION 5

FIRE FIGHTING MEASURES

---

FIRE AND EXPLOSION HAZARD:

Slight fire hazard when exposed to heat or flame.

Dust-air mixtures may ignite or explode.

**EXTINGUISHING MEDIA:**

Dry chemical, carbon dioxide, water spray or regular foam (1990 Emergency Response Guidebook, DOT P 5800.5).

For larger fires, use water spray, fog or regular foam (1990 Emergency Response Guidebook, DOT P 5800.5).

**FIREFIGHTING:**

Move container from fire area if you can do it without risk. Do not scatter spilled material with high-pressure water streams. Dike fire-control water for later disposal (1990 Emergency Response Guidebook, DOT P 5800.5, Guide Page 31).

Use agents suitable for type of surrounding fire. Avoid breathing hazardous vapors, keep upwind.

**HAZARDOUS COMBUSTION PRODUCTS:**

Thermal decomposition products may include toxic oxides of carbon.

---

**SECTION 6****ACCIDENTAL RELEASE MEASURES**

---

**OCCUPATIONAL SPILL:**

Sweep up and place in suitable clean, dry containers for reclamation or later disposal. Do not flush spilled material into sewer. Keep unnecessary people away.

---

**SECTION 7****HANDLING AND STORAGE**

---

Observe all federal, state and local regulations when storing this substance.

Store away from incompatible substances.

Keep in a tightly closed container. Store in a cool, dry, ventilated area.

---

**SECTION 8****EXPOSURE CONTROLS/PERSONAL PROTECTION**

---

**EXPOSURE LIMITS:**

No occupational exposure limits established by OSHA, ACGIH, or NIOSH.

**VENTILATION:**

Provide local exhaust ventilation. Ventilation equipment must be explosion proof.

**EYE PROTECTION:**

Employee must wear splash-proof or dust-resistant safety goggles and a faceshield to prevent contact with this substance.

**Emergency wash facilities:**

Where there is any possibility that an employee's eyes and/or skin may be

exposed to this substance, the employer should provide an eye wash fountain and quick drench shower within the immediate work area for emergency use.

**CLOTHING:**

Employee must wear appropriate protective (impervious) clothing and equipment to prevent repeated or prolonged skin contact with this substance.

**GLOVES:**

Employee must wear appropriate protective gloves to prevent contact with this substance.

**RESPIRATOR:**

The following respirators are recommended based on information found in the physical data, toxicity and health effects sections. They are ranked in order from minimum to maximum respiratory protection.

The specific respirator selected must be based on contamination levels found in the work place, must be based on the specific operation, must not exceed the working limits of the respirator and must be jointly approved by the National Institute for Occupational Safety and Health and the Mine Safety and Health Administration (NIOSH-MSHA).

Any dust and mist respirator with a full facepiece.

Any air-purifying full facepiece respirator with a high-efficiency particulate filter.

Any powered air-purifying respirator with a tight-fitting facepiece and high-efficiency particulate filter.

Any type 'C' supplied-air respirator with a full facepiece operated in pressure-demand or other positive pressure mode or with a full facepiece, helmet or hood operated in continuous-flow mode.

Any self-contained breathing apparatus with a full facepiece operated in pressure-demand or other positive pressure mode.

**FOR FIREFIGHTING AND OTHER IMMEDIATELY DANGEROUS TO LIFE OR HEALTH CONDITIONS:**

Any self-contained breathing apparatus that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode.

Any supplied-air respirator that has a full facepiece and is operated in a pressure-demand or other positive-pressure mode in combination with an auxiliary self-contained breathing apparatus operated in pressure-demand or other positive-pressure mode.

---

**SECTION 9****PHYSICAL AND CHEMICAL PROPERTIES**

---

**DESCRIPTION:** Odorless, colorless, orthorhombic prisms with pleasant, sour taste. Efflorescent in dry air; slightly deliquescent in moist air.

**MOLECULAR WEIGHT:** 210.14

**MOLECULAR FORMULA:** H-O2-C3-H2-(O-H)-C-O2-H-C-H2-C-O2-H.H2-O

MELTING POINT: 212 F (100 C)  
SPECIFIC GRAVITY: 1.542  
WATER SOLUBILITY: complete  
PH: 2.2 @ 0.1 N soln  
SOLVENT SOLUBILITY: Soluble in amyl alcohol, methanol and propanol;  
moderately soluble in amyl acetate, ethyl acetate and ether; very slightly  
soluble in chloroform.

loses water of crystallization at about 104-122 F (40-50 C)

---

## SECTION 10

## STABILITY AND REACTIVITY

---

### REACTIVITY:

Stable under normal temperatures and pressures.

### CONDITIONS TO AVOID:

May burn but does not ignite readily. Avoid contact with strong oxidizers,  
excessive heat, sparks, or open flame.

### INCOMPATIBILITIES:

#### CITRIC ACID:

BASES: Incompatible.

METAL NITRATES: Possible explosion hazard.

OXIDIZERS (STRONG): Fire and explosion hazard.

### HAZARDOUS DECOMPOSITION:

Thermal decomposition products may include toxic oxides of carbon.

### POLYMERIZATION:

Hazardous polymerization has not been reported to occur under normal  
temperatures and pressures.

---

## SECTION 11

## TOXICOLOGY INFORMATION

---

### CITRIC ACID:

#### IRRITATION DATA:

ANHYDROUS: 500 mg/24 hours skin-rabbit mild; 750 ug/24 hours eye-rabbit  
severe.

MONOHYDRATE: 5 mg/30 seconds rinsed eye-rabbit mild.

#### TOXICITY DATA:

ANHYDROUS: 5040 mg/kg oral-mouse LD50; 7000 mg/kg oral-rabbit LDLo;

42 mg/kg intravenous-mouse LD50; 330 mg/kg intravenous-rabbit LD50;

883 mg/kg intraperitoneal-rat LD50; 903 mg/kg intraperitoneal-mouse LD50;

5500 mg/kg subcutaneous-rat LD50; 2700 mg/kg subcutaneous-mouse LD50.

MONOHYDRATE: 375 mg/kg intraperitoneal-rat LD50.

#### CARCINOGEN STATUS: None.

LOCAL EFFECTS: Corrosive- eye; irritant- inhalation, skin.

ACUTE TOXICITY LEVEL: Slightly toxic by ingestion (anhydrous).

AT INCREASED RISK FROM EXPOSURE: Persons with renal impairment, Addison's  
disease, dehydration, or hyperkalemia.

### HEALTH EFFECTS

INHALATION:  
CITRIC ACID:  
IRRITANT.

ACUTE EXPOSURE- Inhalation may cause mucous membrane irritation with sore throat, coughing and shortness of breath. Allergic reactions may occur in some individuals.

CHRONIC EXPOSURE- Long-term overexposure may damage tooth enamel.

SKIN CONTACT:  
CITRIC ACID:  
IRRITANT.

ACUTE EXPOSURE- Contact may cause moderate irritation with redness and pain. Allergic reactions may occur in some individuals.

CHRONIC EXPOSURE- Repeated or prolonged contact with irritants may cause dermatitis.

EYE CONTACT:  
CITRIC ACID:  
CORROSIVE.

ACUTE EXPOSURE- Contact may cause severe irritation with redness, pain and possibly burns. A large quantity of saturated solution splashed in human eyes produced a severe conjunctival reaction and ulceration of the cornea, resulting in extensive adherent leukoma. Irrigation with a 0.5% solution for 30 minutes caused permanent cloudiness of the cornea in rabbit eyes; a 2% solution caused severe dense opacification.

CHRONIC EXPOSURE- Effects are dependent upon concentration and duration of exposure. Conjunctivitis or effects similar to those as for acute exposure may occur.

INGESTION:  
CITRIC ACID:

ACUTE EXPOSURE- Ingestion of large amounts may cause acute but transient gastrointestinal irritation with vomiting and diarrhea. Concentrated solutions may cause mild corrosion of the upper gastrointestinal tract with sore throat, abdominal pain, wrinkling and roughening of mucous membranes of the mouth and esophagus, and swelling and discoloration of the stomach and intestine. A human death has been reported following ingestion of 20-30 grams of the pure acid. Lethal doses in rats produced metabolic acidosis and calcium deficiency.

CHRONIC EXPOSURE- Frequent or excessive intake of citric acid may cause erosion of the teeth and local irritation. Prolonged feeding studies in animals resulted only in a slight increase in dental attrition.

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SECTION 12

ECOLOGICAL INFORMATION

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ENVIRONMENTAL IMPACT RATING (0-4): no data available

ACUTE AQUATIC TOXICITY: no data available

DEGRADABILITY: no data available

LOG BIOCONCENTRATION FACTOR (BCF): no data available

LOG OCTANOL/WATER PARTITION COEFFICIENT: no data available

---

SECTION 13DISPOSAL INFORMATION

---

Observe all federal, state and local regulations when disposing of this substance.

---

SECTION 14TRANSPORTATION INFORMATION

---

No classification currently assigned

---

SECTION 15REGULATORY INFORMATION

---

TSCA STATUS: N

CERCLA SECTION 103 (40CFR302.4):	N
SARA SECTION 302 (40CFR355.30):	N
SARA SECTION 304 (40CFR355.40):	N
SARA SECTION 313 (40CFR372.65):	N
OSHA PROCESS SAFETY (29CFR1910.119):	N
CALIFORNIA PROPOSITION 65:	N

SARA HAZARD CATEGORIES, SARA SECTIONS 311/312 (40 CFR 370.21)

ACUTE HAZARD:	Y
CHRONIC HAZARD:	N
FIRE HAZARD:	N
REACTIVITY HAZARD:	N
SUDDEN RELEASE HAZARD:	N

---

SECTION 16OTHER

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JHS14480

PAGE 8

NON-BULK PACKAGING: 49 CFR 173.203  
BULK PACKAGING: 49 CFR 173.241

U.S. DEPARTMENT OF TRANSPORTATION QUANTITY LIMITATIONS 49 CFR 172.101:  
PASSENGER AIRCRAFT OR RAILCAR: 60 1  
CARGO AIRCRAFT ONLY: 220 1

-----  
SECTION 15REGULATORY INFORMATION  
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TSCA STATUS: Y

CERCLA SECTION 103 (40CFR302.4): N  
SARA SECTION 302 (40CFR355.30): N  
SARA SECTION 304 (40CFR355.40): N  
SARA SECTION 313 (40CFR372.65): N  
OSHA PROCESS SAFETY (29CFR1910.119): N  
CALIFORNIA PROPOSITION 65: N

SARA HAZARD CATEGORIES, SARA SECTIONS 311/312 (40 CFR 370.21)

ACUTE HAZARD: Y  
CHRONIC HAZARD: N  
FIRE HAZARD: Y  
REACTIVITY HAZARD: N  
SUDDEN RELEASE HAZARD: N

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SECTION 16OTHER  
-----

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## UNION CARBIDE CHEMICALS AND PLASTICS COMPANY INC.

Industrial Chemicals Division  
MATERIAL SAFETY DATA SHEET

EFFECTIVE DATE 10/21/93

Union Carbide urges each customer or recipient of this MSDS to study it carefully to become aware of and understand the hazards associated with the product. The reader should consider consulting reference works or individuals who are experts in ventilation, toxicology, and fire prevention, as necessary or appropriate to use and understand the data contained in this MSDS.

To promote safe handling, each customer or recipient should: (1) notify its employees, agents, contractors and others whom it knows or believes will use this material or the information in this MSDS and any other information regarding hazards or safety; (2) furnish this same information to each of its customers for the product; and (3) request its customers to notify their employees, customers, and other users of the product of this information.

## I. IDENTIFICATION

PRODUCT NAME: TRITON-RW-50 Surfactant

CHEMICAL NAME: Amine Ethoxylate

CHEMICAL FAMILY: Surfactant

FORMULA: Not Applicable

MOLECULAR WEIGHT: Not Applicable

SYNONYMS: Nonionic Polyethoxy Amine Surfactant

CAS # AND NAME: 73138-27-9  
Amines, C12-14-tert-alkyl, ethoxylated

## II. PHYSICAL DATA (Determined on Typical Material)

BOILING POINT, 760 mm.Hg: 269.9°C (517.8°F)

SPECIFIC GRAVITY(H<sub>2</sub>O = 1): 0.993 at 20/20°C

FREEZING POINT: Pour point  
-27°C (-17°F)

VAPOR PRESSURE AT 20°C: <0.01 mmHg

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EMERGENCY PHONE NUMBERS: 1-800-UCC-HELP (NUMBER AVAILABLE AT ALL TIMES) OR (304) 744-3487

UNION CARBIDE CHEMICALS AND PLASTICS COMPANY INC  
Industrial Chemicals Division  
39 Old Ridgebury Road, Danbury, CT 06817-0001



PRODUCT NAME: TRITON RW-50 Surfactant<sup>B-36</sup>

EVAPORATION RATE (Butyl Acetate = 1): &lt;0.01

VAPOR DENSITY (AIR = 1): &gt;10

SOLUBILITY IN WATER by wt: 100% at 20°C

APPEARANCE: Transparent dark yellow

ODOR: Pungent

PHYSICAL STATE: Liquid

PERCENT VOLATILES (by weight): 0.5

### III. INGREDIENTS

<u>%</u>	<u>MATERIAL</u>	<u>CAS#</u>	<u>EXPOSURE LIMIT</u>
> 97	C12-14 t-alkylamines, ethoxylated	73138-27-9	None established
< 0.5	C12-14 t-alkylamines	68955-53-3	None established
< 3	Polyethylene Glycol	25322-68-3	None established

### IV. FIRE AND EXPLOSION HAZARD DATA

FLASH POINT(test method(s)): 310°F (154°C)  
Pensky-Martens Closed Cup ASTM D 93  
425°F (218°C)  
Cleveland Open Cup ASTM D 92

FLAMMABLE LIMITS IN AIR LOWER: Not Determined  
% by volume: UPPER: Not Determined

#### SPECIAL FIRE FIGHTING PROCEDURES:

Do not direct a solid stream of water or foam into hot, burning pools; this may cause frothing and increase fire intensity.  
Use self-contained breathing apparatus and protective clothing.

#### EXTINGUISHING MEDIA:

Apply alcohol-type or all-purpose-type foam by manufacturers' recommended techniques for large fires. Use water spray, carbon dioxide, or dry chemical media for small fires.

---

PRODUCT NAME: TRITON RW-50 Surfactant B-37

---

UNUSUAL FIRE AND  
EXPLOSION HAZARDS:

This material may produce a floating fire hazard.  
During a fire, nitrogen oxides and alkylamines may be produced.

---

V. HEALTH HAZARD DATA

---

EXPOSURE LIMIT(S):

None established by ACGIH or OSHA.

---

EFFECTS OF SINGLE OVEREXPOSURE:

---

SWALLOWING:

Moderately toxic. May cause irritation of the mouth, throat, esophagus and stomach with nausea, abdominal discomfort, vomiting and diarrhea. Aspiration into the lungs may occur during ingestion or vomiting, resulting in lung injury.

---

SKIN ABSORPTION:

Prolonged or widespread contact may result in the absorption of potentially harmful amounts of material.

---

INHALATION:

High concentrations of vapor or mist cause irritation of the respiratory tract, experienced as nasal discomfort and discharge, with chest pain and coughing.

---

SKIN CONTACT:

Causes irritation with discomfort, local redness, and possible swelling.

---

EYE CONTACT:

Causes severe irritation, experienced as discomfort or pain, excess blinking and tear production, marked excess redness and swelling of the conjunctiva, and chemical burns of the eye.

---

EFFECTS OF REPEATED OVEREXPOSURE:

No adverse effects anticipated from available information.

---

MEDICAL CONDITIONS AGGRAVATED BY OVEREXPOSURE:

Skin contact may aggravate an existing dermatitis.

---

SIGNIFICANT LABORATORY DATA WITH POSSIBLE RELEVANCE TO HUMAN  
HEALTH HAZARD EVALUATION:

Contains surfactant which, based on studies with rabbits involving the sustained occluded contact of the undiluted surfactant with skin, indicate that such conditions may result in the development of inflammatory changes in the lung.

---

OTHER EFFECTS OF OVEREXPOSURE:

Because of its irritant and surfactant properties, breathing vapor or mist of this material, as might be generated in spraying or heating applications, may result in lung injury similar to that observed following exposure by direct deposition of surfactant into the lung.

---

EMERGENCY AND FIRST AID PROCEDURES:

---

SWALLOWING:

If patient is fully conscious, give two glasses of water at once. Do not

PRODUCT NAME: TRITON RW-50 Surfactant B-38

---

induce vomiting. Obtain medical attention.

---

SKIN: Remove contaminated clothing. Wash skin with soap and water. If irritation persists or if contact has been prolonged, obtain medical attention.

---

INHALATION: Remove to fresh air. Obtain medical attention if symptoms persist.

---

EYES: Immediately flush eyes with water and continue washing for at least 15 minutes. Obtain medical attention without delay, preferably from an ophthalmologist.

---

NOTES TO PHYSICIAN: There is no specific antidote. Treatment of overexposure should be directed at the control of symptoms and the clinical condition of the patient. Any material aspirated during vomiting may cause lung injury. Therefore, emesis should not be induced mechanically or pharmacologically. If it is considered necessary to evacuate the stomach contents, this should be done by means least likely to cause aspiration (e.g., gastric lavage after endotracheal intubation).

---

## VI. REACTIVITY DATA

---

STABILITY: Stable

---

CONDITIONS TO AVOID: Prolonged excessive heat may cause product decomposition.

---

INCOMPATIBILITY (materials to avoid):

Avoid contact with strong oxidizing and/or reducing agents. Avoid strong bases at high temperatures, strong acids, and materials reactive with amines and hydroxyl compounds.

---

HAZARDOUS COMBUSTION OR DECOMPOSITION PRODUCTS:

Thermal decomposition may yield alkylamines. Burning can produce carbon monoxide and/or carbon dioxide, and nitrogen oxides.

---

HAZARDOUS POLYMERIZATION: Will Not Occur

---

CONDITIONS TO AVOID: None known.

---

## VII. SPILL OR LEAK PROCEDURES

---

STEPS TO BE TAKEN IF MATERIAL IS RELEASED OR SPILLED:

Wear eye and skin protection. Floor may be slippery; use care to avoid falling. Contain spills immediately with inert materials (eg. sand, earth). Avoid discharge to natural waters. Transfer liquids and solid diking material to suitable containers for recovery or disposal. To avoid gelling and foaming problems, do not use water to flush away spills.

AQUATIC EFFECTS:

48 hr. "Daphnia magna" LC50 - 54 mg/L  
96 hr. Fathead Minnow LC50 - 21 mg/L

---

WASTE DISPOSAL METHOD:

Incinerate in a furnace where permitted under Federal, State, and local regulations.

PRODUCT NAME: TRITON RW-50 Surfactant B-39

---

### VIII. SPECIAL PROTECTION INFORMATION

---

**RESPIRATORY PROTECTION (specify type):**

None expected to be needed. However, where misting may occur, wear a MSHA/NIOSH approved (or equivalent) half-mask air purifying respirator.

**VENTILATION:**

General (mechanical) room ventilation is expected to be satisfactory.

**PROTECTIVE GLOVES:**

Neoprene, butyl rubber

**EYE PROTECTION:**

Chemical splash goggles and face shield

**OTHER PROTECTIVE EQUIPMENT:**

Eye Bath, Safety Shower  
Full Protective Clothing

---

### IX. SPECIAL PRECAUTIONS

---

**PRECAUTIONS TO BE TAKEN IN HANDLING AND STORAGE:**

DANGER! Causes eye burns.  
Causes skin irritation.  
Aspiration may cause lung damage.  
Do not get in eyes.  
Avoid contact with skin and clothing.  
Do not swallow.  
Keep container closed.  
Use with adequate ventilation.  
Wash thoroughly after handling.

FOR INDUSTRY USE ONLY

**OTHER PRECAUTIONS:**

Surfactants can cause foaming problems in biological wastewater treatment plants and other high shear operations.

This product may contain trace amounts of Ethylene Oxide (CAS No. 75-21-8), a condition which creates the potential for accumulation of Ethylene Oxide in the head space of shipping and storage containers and in enclosed areas where the product is being handled or used. Ethylene Oxide is considered by OSHA, IARC, and NTP as a potential carcinogen for humans. OSHA considers that, at excess levels, Ethylene Oxide may present reproductive, mutagenic, genotoxic, neurologic and sensitization hazards in humans. If this product is handled with adequate ventilation, the presence of these trace amounts is not expected to result in any short or long term hazard.

This product may not be exempt from OSHA'S Ethylene Oxide standard, 29CFR1910.1047. Users should comply with all applicable provisions. Personnel should be monitored to determine levels of exposure to Ethylene Oxide. If necessary, protective measures should be taken. The OSHA permissible exposure limit for Ethylene Oxide is 1 ppm TWA8, the action level is 0.5 ppm TWA8, the ACGIH TLV is 1 ppm TWA8 and OSHA has established an excursion limit of 5 ppm (15 minute average).

WARNING: Sudden release of hot organic chemical vapors or mists from process equipment operating at elevated temperature and pressure, or sudden ingress of air into vacuum equipment, may result in ignitions without the presence of obvious ignition sources. Published "autoignition" or

PRODUCT NAME: TRITON RW-50 Surfactant B-40

"ignition" temperature values cannot be treated as safe operating temperatures in chemical processes without analysis of the actual process conditions.

Any use of this product in elevated-temperature processes should be thoroughly evaluated to establish and maintain safe operating conditions. Further information is available in a technical bulletin entitled "Ignition Hazards of Organic Chemical Vapors."

## X. REGULATORY INFORMATION

### STATUS ON SUBSTANCE LISTS:

The concentrations shown are maximum or ceiling levels (weight %) to be used for calculations for regulations. Trade Secrets are indicated by "TS".

#### FEDERAL EPA

Comprehensive Environmental Response Compensation, and Liability Act of 1980 (CERCLA) requires notification of the National Response Center of release of quantities of Hazardous Substances equal to or greater than the reportable quantities (RQs) in 40 CFR 302.4.

Components present in this product at a level which could require reporting under the statute are:

CHEMICAL	UPPER BOUND	
	CAS NUMBER	CONCENTRATION %
Dioxane	123-91-1	.0150
Ethylene Oxide	75-21-8	.0010

Superfund Amendments and Reauthorization Act of 1986 (SARA) Title III requires emergency planning based on Threshold Planning Quantities (TPQs) and release reporting based on Reportable Quantities (RQs) in 40 CFR 355 (used for SARA 302, 304, 311 and 312).

Components present in this product at a level which could require reporting under the statute are:

\*\*\*NONE\*\*\*

Superfund Amendments and Reauthorization Act of 1986 (SARA) Title III requires submission of annual reports of release of toxic chemicals that appear in 40 CFR 372 (for SARA 313). This information must be included in all MSDSs that are copied and distributed for this material.

Components present in this product at a level which could require reporting under the statute are:

\*\*\*NONE\*\*\*

### Toxic Substances Control Act (TSCA) STATUS:

The ingredients of this product are on the TSCA inventory.

#### STATE RIGHT-TO-KNOW

#### CALIFORNIA Proposition 65

This product contains trace levels of Dioxane and Ethylene Oxide which the State of California has found to cause cancer, birth defects or other reproductive harm.

MASSACHUSETTS Right-To-Know, Substance List (MSL) Hazardous Substances and Extraordinarily

**PRODUCT NAME: TRITON RW-50 Surfactant B-41**

Hazardous Substances on the MSL must be identified when present in products.

Components present in this product at a level which could require reporting under the statute are:

EXTRAORDINARILY HAZARDOUS SUBSTANCES (  $\geq$  0.0001%)

UPPER BOUND

CHEMICAL	CAS NUMBER	CONCENTRATION %
Dioxane	123-91-1	.0150
Ethylene Oxide	75-21-8	.0010

PENNSYLVANIA Right-to-Know, Hazardous Substance List Hazardous Substances and Special Hazardous Substances on the List must be identified when present in products.

Components present in this product at a level which could require reporting under the statute are:

\*\*\* NONE \*\*\*

CALIFORNIA SCAQMD RULE 443.1 VOC'S:

Vapor Pressure  $< 0.01$  mmHg at 20°C

VOC 5 g/L

VOC 5 g/L less water and less exempted solvents

OTHER REGULATORY INFORMATION:

\*\*\* None known \*\*\*

NOTE ----

The opinions expressed herein are those of qualified experts within Union Carbide. We believe that the information contained herein is current as of the date of this Material Safety Data Sheet. Since the use of this information and the conditions of the use of the product are not under the control of Union Carbide, it is the user's obligation to determine conditions of safe use of the product.

REVISED SECTIONS:

Revisions occurred in the following sections:

Section II - Physical Data

Section X - VOC's have been changed

PRODUCT: 89569

F NUMBER: N0398B